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INORGANIC ION EXCHANGE MEMBRANE FUEL CELL

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INORGANIC ION EXCHANGE MEMBRANE
FUEL CELL

by

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G. Belfort

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

July 1965

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1.0 SUMMARY AND CONCLUSIONS

The emphasis during this report period continued to be on achieving the maximum possible fuel cell performance. By incorporating the optimum fuel cell design features with the optimum membrane-catalyst-electrode configuration, it was possible to achieve the highest fuel cell performance level for the program to date. At 65°C, the voltage was in the 0.770 to 0.780 volt range at 30 ma/cm². At 50 ma/cm², the voltage was in the vicinity of 0.71 volts.

The following is a summary of the more significant results:

(a) The best performing membrane contains 20% platinum black impregnated to a depth of 5% of the total membrane thickness on both sides. This quantity of platinum, constituting about two percent of the total membrane weight, is of the order of 0.07 grams per membrane. A separate tantalum screen—platinum black—Teflon electrode structure is pressed up against this membrane. Evidently the critical reaction zone is near the surface of the membrane. Deeper penetration of the membrane with catalyst material affords no further advantage.

(b) Incorporating 2% Teflon into the outer layer of the membrane as water-proofing agent during the impregnation with platinum black has no apparent effect on the fuel cell performance level at 65°C.

(c) Cycling this fuel cell between 65° and 102°C has no significant effect on fuel cell performance; Teflon does appear to contribute to this stability in performance.

(d) Constant performance levels up to 151°C were obtained for the platinum black impregnated-membrane (penetration of platinum black to a depth of 5% of the total membrane thickness).

(e) Fuel cell performance improved with increased gas free-space in the backup plate, providing there was no loss of total contact area between electrode and backup plate.

(f) A four-inch diameter platinum black-impregnated C200B membrane was successfully evaluated in a new enlarged version of the Astro-power compact fuel cell. The maximum voltage was 0.755 volts at 30 ma/cm² for over 200 hours of continuous operation at 65°C.

(g) It appears that a 50/50 platinum black-palladium black mixture

is a more effective catalyst system for impregnation into the membrane than is either platinum black or palladium black, alone. Studies with the 50/50 mixture are being intensified.

(h) In further efforts to improve fuel cell performance, fifty-mesh tantalum screen electrodes were evaluated by three different methods for incorporating platinum black catalyst into the screen matrix, i. e., electro-deposition, paste and sintering techniques. It was found that all of the techniques gave comparable fuel cell performance. Loading with platinum black catalyst to the extent of 20 to 30 mg/cm² appears to be the range for optimum performance. Silicone resins are about as effective as Teflon for water-proofing purposes in the screen matrix. 304 stainless steel (160 mesh) shows some promise as a potential electrode material for this membrane system.

(i) Unitized membrane-catalyst-electrode system with the three components fused together through sintering have not as yet exhibited significant promising fuel cell behavior. A distinct interface between membrane and electrode surfaces may be required for adequate fuel cell performance; this may be a requirement for the three phase reaction involved.

(j) Fuel cell experiments with provision for wicking are still in progress.

2.0 INTRODUCTION

During the previous quarterly report period, ⁽¹⁾ it was established that, by improved fuel cell design as well as by the use of platinum-impregnated zirconia-phosphoric acid-"Zeolon-H" membranes, a fuel cell performance level of 0.65 volts at 50 ma/cm² (0.72 volts at 30 ma/cm²) could be attained. Then, a new performance target level of 0.7 volts at 50 ma/cm² was established. It was expected that this new goal could be attained by continued improved fuel cell design efforts as well as through more effective membrane-catalyst-electrode configurations. The new performance goal was reached in a fuel cell test under the most favorable operational conditions established to date.

A larger membrane having a diameter of four inches also afforded favorable performance characteristics under essentially similar conditions.

Attention was given to improving the membrane-catalyst-electrode configuration, particularly from the standpoint of reduction of contact resistances. It was found that the use of a 50/50 platinum-palladium black catalyst mixture is a more effective catalyst system than platinum black alone.

The details of this work are presented in this report.

3.0 EXPERIMENTAL PROCEDURE, RESULTS AND DISCUSSION

3.1 Fuel Cell Tests

During this report period, the emphasis continued to be on obtaining optimum fuel cell performance. The results of the most significant tests performed with the C200B membrane and with American Cyanamid Type AA-1 electrodes in the Astropower standard (analytical) fuel cell⁽²⁾ and the compact fuel cell design⁽¹⁾ are compiled in Tables I and II, respectively. Tables III, V and VI depict the various experiments performed in the analytical fuel cell involving different types of catalyst-electrode configurations.

All tests were performed at a constant current density of 30 ma/cm². The results obtained are discussed below.

It is to be noted that fuel cell resistance listed in Tables I - VI inclusive, had been calculated from the slopes of the straight portions of the corresponding polarization curves. Essentially, they represent the ohmic resistance for the cell at that particular point of the test.

3.1.1 Standard Fuel Cell Tests

Test (1), continued from the previous quarterly period,⁽²⁾ was performed at 25°C in order to establish the effect of temperature and membrane thickness on performance. Tests (6), (7) and (8) are in the same category as well. The plots of Figures 1 and 2 compare both the cell voltages and fuel cell resistances as obtained from polarization curves of Test (1) with corresponding values obtained previously for a similar membrane at 65°C, (Test 5, Table I).⁽¹⁾ Evidently, as temperature increases from 25°C to 65°C, fuel cell performance improves. As described previously, we conclude that the higher fuel cell resistance at 25°C is due to inadequate product water removal resulting from lower vaporization at this temperature. Additional waterproofing at the membrane-electrode interface should improve the performance at that temperature. However, Test (1) does demonstrate stable operational feasibility at 25°C for the C200B membrane. As far as the membrane thickness is concerned, membrane thickness in the range of 0.50 to 0.75 mm does not appear to affect fuel cell performance.⁽¹⁾

It was shown in Reference 1 that optimum performance

occurred within the temperature range of 65° to 120°C. Hence, fuel cell tests were performed at 65°C in further efforts to ascertain the maximum possible level of performance.

Test (2) was run with platinum black impregnated in the membrane to the extent of one-third the thickness of the membrane, as described in Reference 1. The concentration of platinum in both outer one-third layers was 20% by weight and the middle layer was comprised of C200B material alone. This amounts to about 0.5 grams platinum per membrane which is about 14% of the total membrane weight. Actually, Test (2) concludes the study conducted at 65°C, described in Reference 1, wherein the concentration of platinum black in the outer one-third layers of the membrane was varied from 10% up to 40% by weight. The results are summarized in Figures 3 and 4. In Figure 3 the voltage-time curves are plotted and the corresponding resistance-time curves are given in Figure 4. Both plots indicate that in the case of 10% platinum black impregnation, poorer performance results, whereas performance remains essentially constant over the 20 to 40% range. Although impregnating platinum black into the outer one-third layers of the C200B membrane did not afford significantly higher performance than the untreated membranes at 65°C, ⁽¹⁾ it accounts for stable and essentially constant performance at temperatures as high as 151°C. To date untreated C200B membranes have manifested favorable fuel cell performance levels at temperatures higher than 75°C, which have been of shorter duration.

In Reference 1 it was observed that the active area for electrocatalysis appears to be at the surface of the membrane only. As a result, the membrane used in Test (3) had been impregnated only to the extent of about 5% on both sides, with 20% platinum black by weight, leaving the middle 90% layer with nonimpregnated C200B material. This amounts to about 0.07 grams of platinum per membrane which is about 2% of the total membrane weight. Test (4) was performed with 2% Teflon by weight incorporated in the two outer one-twentieth layers containing 20% by weight of platinum black. No significant performance improvement was observed by adding the 2% Teflon waterproofing agent. This experiment will be attempted at 25°C in order to ascertain whether the additional amount of waterproofing agent enhances performance at that temperature. Test (6) was run to determine further whether fuel cell performance varied over the temperature range of

65° to 120°C; it had Teflon incorporated into the system as well. In this test only a fine spray, resulting in a light surface coating, was administered to the membrane surface. The composite membrane sandwich was re-sintered at 300°C prior to the test. The results of this test indicate that constant, stable performance is possible for this membrane system over the temperature cycle of 65° to 102° and back to 65°C. Teflon contributes to the stability in performance.

Test (5) was the first attempt to incorporate back-up plates with 136 holes and groove width of 0.105 inch in the old (analytical) fuel cell. Although the initial performance looked good and a high performance was expected, the nature of the "Zeolon-H" as supplied by the Norton Company resulted in significantly weaker membrane structures and consequently, poor fuel cell performance. This matter was discussed in Reference 3.

Tests (7) and (8) were run at 70°C and 151°C, respectively. The purpose of both tests was to observe the fuel cell performances of the C200B membrane impregnated with platinum black to the extent of 20% in the outer one-twentieth layers, (0.07 grams platinum per membrane) at elevated temperatures. Resistances obtained from polarization curves in Tests (7) and (8) are similar, demonstrating that fuel cell performance does not vary with temperature in the range of 65° to 151°C. As discussed in Reference 1, the constancy of performance with increase in temperature of the Astropower inorganic membrane fuel cell is due to the invariance of the ionic conductivity of the membrane with rise in temperature, at least to any significant extent. Electrode kinetics do not appear to be controlling up to 151°C.

Polarization curves for Tests (2), (3), (4), (6), (7) and (8) are given in Figures 5 through 10.

3.1.2 Compact Fuel Cell Life Tests

Most of the work done with the Astropower compact fuel cell involved two units requiring a two-inch diameter membrane. This design can be seen in Figure 3, Reference 1. The results obtained for tests depicting the most significant results are given in Table II. This fuel cell was designed using mass and heat transfer analysis and engineering experience, as discussed in Reference 1.

The purpose of Test (9) was to determine whether contact resistance between electrode and backup plate could be decreased by the gold-surfacing of the stainless steel backup plate. This was done by a commercial electrodeposition process which provided a gold deposit 5.0×10^{-5} to 10.0×10^{-5} inch thick. The results of Test (9) show that no enhancement in performance occurs.

It was shown in Reference 1 that the free gas space and the number of holes in the backup plate were directly related. As indicated at that point, it would be expected that increased free gas space should lead to more reaction sites and consequently better performance, provided the amount of metal to metal contact between electrode and backup plate is not substantially reduced. As a result, Test (10) was performed with backup plates having 136 one-eighth inch diameter holes with groove widths of one-eighth inch. Apparently, in Test (10), there is no enhancement over the previous optimum performance which used backup plates with 96 similarly sized holes, because in order to provide for more holes in the backup plates, it was necessary to reduce the size of the contact areas (lugs) on the electrode side of the backup plates. Hence, the next step was to increase the metal to metal contact between the backup plate and electrode, and yet retain the 136 one-eighth inch diameter holes. To accomplish this, the electrode side of the backup plates was modified by decreasing the channel groove width from 0.125 to 0.105 inch. Figure 11 shows how this modification increases the lug size. Figure 13 shows the crisscross arrangement of channel grooves on the electrode facing side for the three types of backup plates, each with a different number of holes. The alignments of grooves and lugs are apparent.

Test 11 involved the use of this apparently improved backup plate modification. The level of performance was the highest obtained to date in the program. Correspondingly, periodic polarization curves gave the lowest fuel cell resistances (0.18 to 0.20 ohms) obtained to date. Figure 14 shows an extension of the plot originally given in Reference 1, describing the relationship between gas exposed area and backup plate design. Hence, if both gas exposed area and lug contact area are maximized simultaneously, the best possible performance for this type of configuration should result. Figure 15 relates gas exposed area and the lowest fuel cell resistance obtained for each backup plate shown in Figure 14. The flattening out of the curve indicates that the optimum gas exposed area and lug-contact area are being approached. Figure 16

shows the impression that the backup plate contact lugs make with the AA-1 Type, American Cyanamid electrode. It can be seen that the area exposed to the gas is considerable, while the electrical contact points are evenly distributed.

The purpose of Test (12) was to compare the performance of the fuel cell when the electrical leads were connected to projecting tabs cut out of the American Cyanamid AA-1 electrodes. Ordinarily, the electrical leads are connected to the backup plates. As can be seen from the polarization curves in Figure 23, the tabs offer considerably higher resistance than do the backup plates. It is quite possible that the IR drop laterally across the electrodes contributes to this enhanced resistance.

With a promising start, it was thought that Test (13) would duplicate the maximum performance, but the cell performance diminished considerably after 104 hours. As mentioned previously in Test (5), the failure could be the result of irregular "Zeolon-H" received from the vendor which caused significantly weaker membranes.

Tests (14) and (15) were both evaluated in a large diameter compact fuel cell using a four-inch diameter membrane. This unit is shown in Figure 19. Test (14) used a C200B membrane with no special treatment while Test (15) had a C200B membrane impregnated with platinum black to the extent of 20% in the outer one-twentieth layers. It was believed that the relatively low current density obtained in Test (14) after 74 hours of operation at 65°C was due to inadequate distribution of reactant gases over the entire area of the backup plate. Therefore, gas diffuser plates were installed in both compartments in Test (15), as illustrated in Figure 18. At a current density of 30 ma/cm², comparable results were obtained in Test (15) as in the two-inch standard or compact fuel cell tests. This demonstrates that a four-fold increase in the area has little or no effect on the fuel cell performance. Figures 20 through 25 are polarization curves for Tests (9), (10), (11), (12), (14) and (15).

Experiments involving a wicking arrangement using cellulosic felt are being continued and the results are being analyzed. A complete description of this work will be presented in ensuing reports.

3.1.3 Fuel Cell Tests with Palladium and Iridium Catalyst Material

Tests were performed in the analytical fuel cell with C200B-catalyst composite membranes prepared as described in Reference 1. These membranes consisted of three layers of compressed material. The two outer layers were composed of mixtures of C200B membrane material and catalyst; the center section consisted of C200B material alone. In this study, the effectiveness of 50/50 platinum-palladium black mixtures simultaneously on both the hydrogen and oxygen sides, 50/50 platinum-iridium black mixtures on the hydrogen side only, and 50/50 silver-palladium black mixtures on the oxygen side only were determined. The work was performed with American-Cyanamid Type AA-1 electrodes, the standard for fuel cell tests on this program. Additional quantities of the appropriate catalyst mixture (0.025 g/cm^2) were sprinkled on each side of the membrane during the electrode-membrane assembling.

The results obtained are summarized in Table III. Noteworthy performance was obtained for the 50/50 platinum-palladium catalyst mixture (Tests 16 and 17). The fuel cell resistance of this catalyst-membrane system, as determined from polarization data, is less than that obtained when either platinum or palladium catalysts are used separately. This is indicated by the summary of resistances obtained from fuel cell studies performed under comparable conditions given in Table IV. The capability of the 50/50 platinum-palladium catalyst combination merits further study.

Referring back to Table III, it is evident that the platinum-iridium catalyst combination on the hydrogen side and the silver-palladium combination for the oxygen electrode show no promise for this application.

3.1.4 Fuel Cell Tests with Various Types of Tantalum and Stainless Screen Electrodes

The purpose of this effort was to ascertain the relative merits of tantalum electrodes having platinum incorporated by electrodeposition, paste and sintering techniques. In addition, it was desired to ascertain the effects of varying amounts of Teflon and silicone waterproofing agents. Fifty mesh tantalum screens were used throughout this study.

Electrodeposition was carried out in 3% H_2PtCl_6 (aq). Up to 40% (by weight of screen) of platinum was deposited in this fashion. The pastes consisted of platinum black suspended in a toluene solution of silicone resin (G.E. SR-224). After application of the paste to the screen, the toluene was evaporated and the resin was cured at 90°C . In addition, pastes were made of platinum black in aqueous Teflon suspension. Sintered electrodes were prepared in the following manner. First, a thin film of platinum black was electrodeposited on the tantalum screen from 3% H_2PtCl_6 (aq). solution. Then, the screens were subjected to three or four sintering cycles in which they were successively immersed in an aqueous suspension of Teflon, dusted with finely divided platinum black, lightly pressed between two sheets of aluminum foil, and sintered for two minutes at 350°C . The amount of Teflon contained in the finished electrodes was estimated to be 5% by weight. The concentration of platinum was varied from 15 mg/cm^2 up to 30 mg/cm^2 .

The results obtained with the standard C200B membrane are summarized in Table V and the results obtained with the C200B-platinum impregnated membranes are summarized in Table VI. Certain trends are evident in Table V, i. e. ,

- (a) The different techniques for platinum catalyst impregnation are essentially equally effective. (Similar conclusions were reached by General Electric.)⁽⁴⁾
- (b) Increasing amounts of platinum catalyst up to as high as 20 to 30 mg/cm^2 are conducive to improved fuel cell performance.
- (c) Silicones appear to be as effective as Teflon as waterproofing agents.

The results of Table VI indicate that the above trends are applicable as well. In addition, it appears that a platinum loading as high as 40 mg/cm^2 is deleterious to fuel cell performance. Possibly, the optimum range for platinum loading is 20 to 30 mg/cm^2 . This matter is being determined at the present time. Optical transmission measurements on the various experimental electrode screens indicated a considerable decrease in

electrode porosity when the catalyst loading was as high as 40 mg/cm^2 . That reasonably good performance was obtained with the stainless steel electrode screen (Test 31) in this initial exploratory study, merits further attention since stainless steel is considerably less expensive and more readily available than platinum black.

3.1.5 Fuel Cell Tests with Unitized Membrane-Catalyst-Electrode Structures

Two types of structures have been prepared and evaluated in a fuel cell, without particularly worthwhile results being achieved in either case. In one type of structure, electrode materials such as platinum, palladium and tantalum in their resinate solutions have been silk screened on the surfaces of platinum-impregnated membrane composites both before and after their final sintering (referred to as diffusion bonding in Reference 1). Then, the entire assembly was fired to bond the electrode layers to the catalyst-membrane composite.

Figure 26 is a photograph of the front view of such a configuration. The fact that significant improvement in performance was obtained when the usual platinum-Teflon-tantalum screen gas diffusion electrode was returned to the same fuel cell in one particular experiment, indicates that adequate electrocatalysis was not achieved by the silk screening approach.

In the second approach the waterproofed, platinized tantalum screen electrode, together with its tantalum leads were pressed and sintered directly into the membrane structure. The system was used with backup plates acting as a support for the unitized structure. A photograph of this structure is Figure 27. Possibly, a distinct interface between membrane and electrode is a requirement for the proper functioning of the inorganic membrane fuel cell. The best results achieved with this type of membrane-catalyst-electrode structure to date have been 1 ma/cm^2 at 0.3 volts and 65°C . This matter will be given further attention.

4.0 FUTURE WORK

(1) The study of catalyst-membrane composites and electrode-catalyst-membrane composites according to the plan given in Reference 1 will be completed. Particular attention will be given to the 50/50 platinum-palladium catalyst mixture incorporated in both the membrane and porous electrode structure.

(2) Ruthenium and rhodium will be investigated as catalyst material.

(3) The aspect of ohmic resistance contribution to electrode polarization will be determined by current-interruptor experiments. This will be correlated with previously determined membrane resistivities at 1000 cps. Efforts will be made to minimize contact resistance to the fullest extent.

(4) The influence of wicking arrangements on fuel cell performance will be established.

5.0 REFERENCES

- (1) "Inorganic Ion Exchange Membrane Fuel Cell," NASA-Lewis Research Center Contract NAS 3-6000, Quarterly Progress Report SM-46221-Q3, Period Ending 10 April 1965.
- (2) Ibid., Quarterly Progress Report SM-46221-Q2, Period Ending 10 January 1965.
- (3) Ibid., Monthly Progress Report for Period Ending 10 June 1965. Report SM-46221-M11.
- (4) "Research on Low Temperature Fuel Cell Systems," Contract DA-44-009-Eng-3771, U. S. Army Research and Development Laboratories, Ft. Belvoir, Virginia, 1961.

6.0 PROJECT PERSONNEL

The following Astropower staff personnel were connected with the program during the reporting period.

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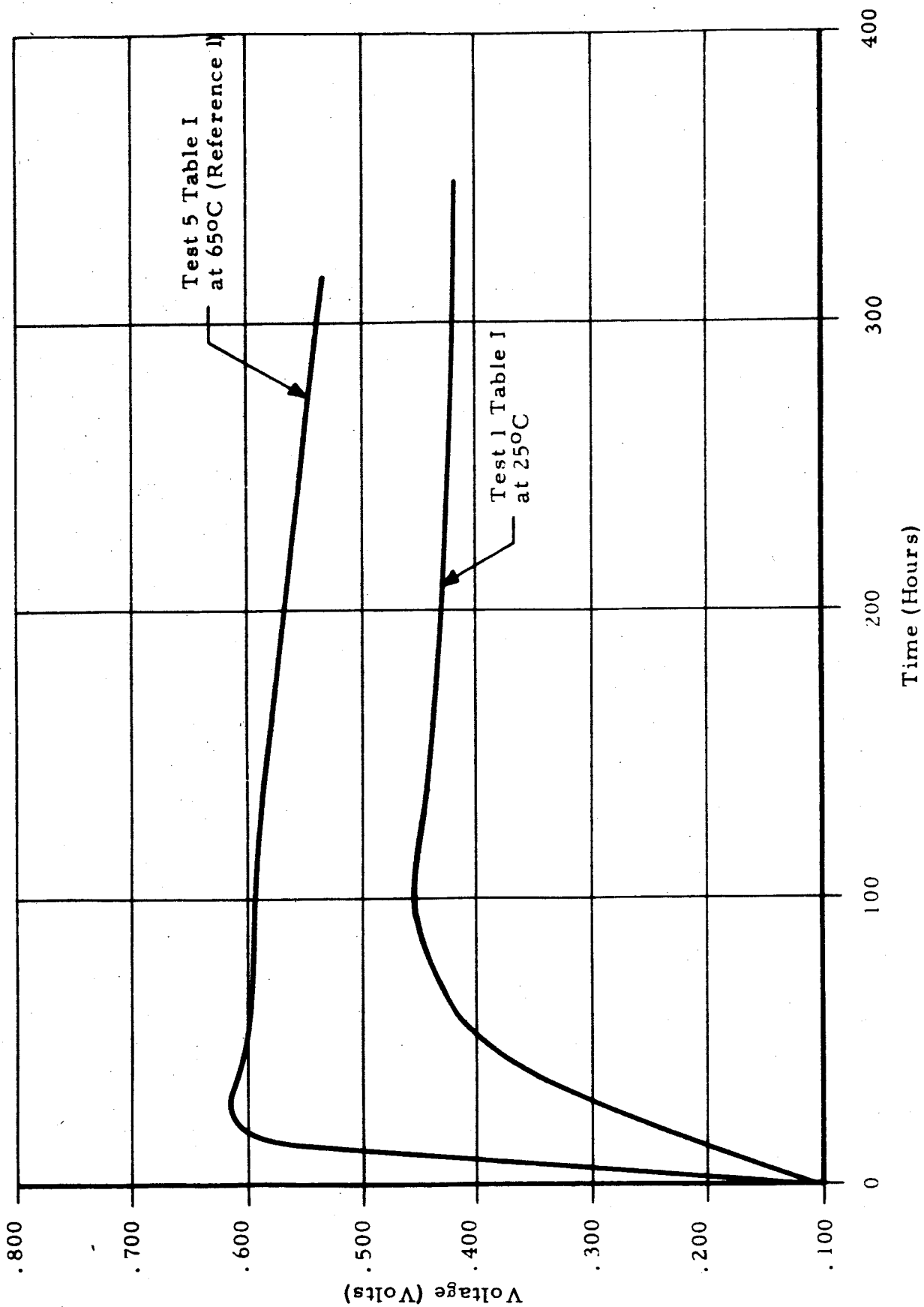


Figure 1. Plot of Voltage versus Time for Fuel Cell Tests at 25°C and 65°C for Non-Impregnated C200B Membrane at 30 ma/cm². (Add 0.03 volts for leads correction)

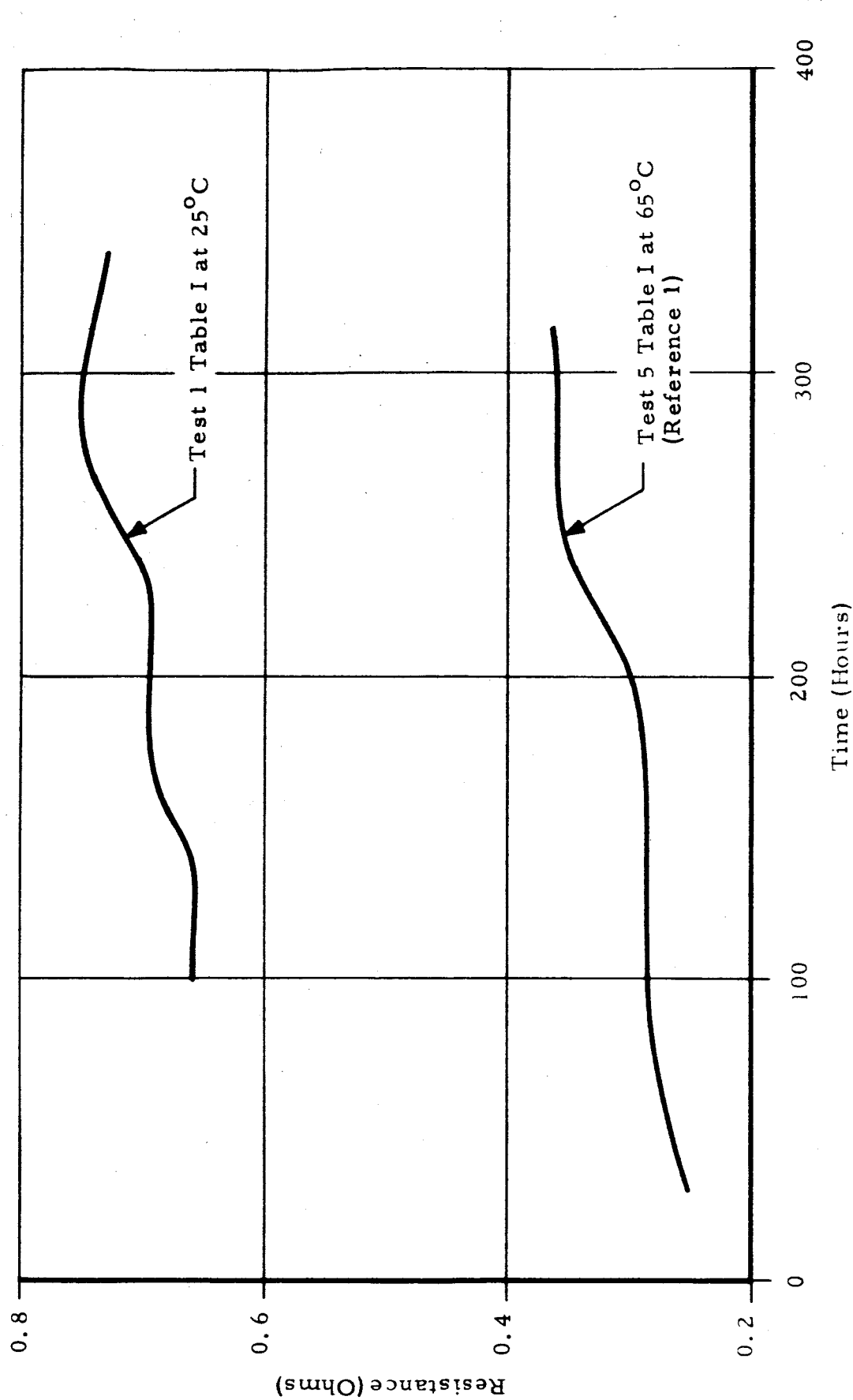
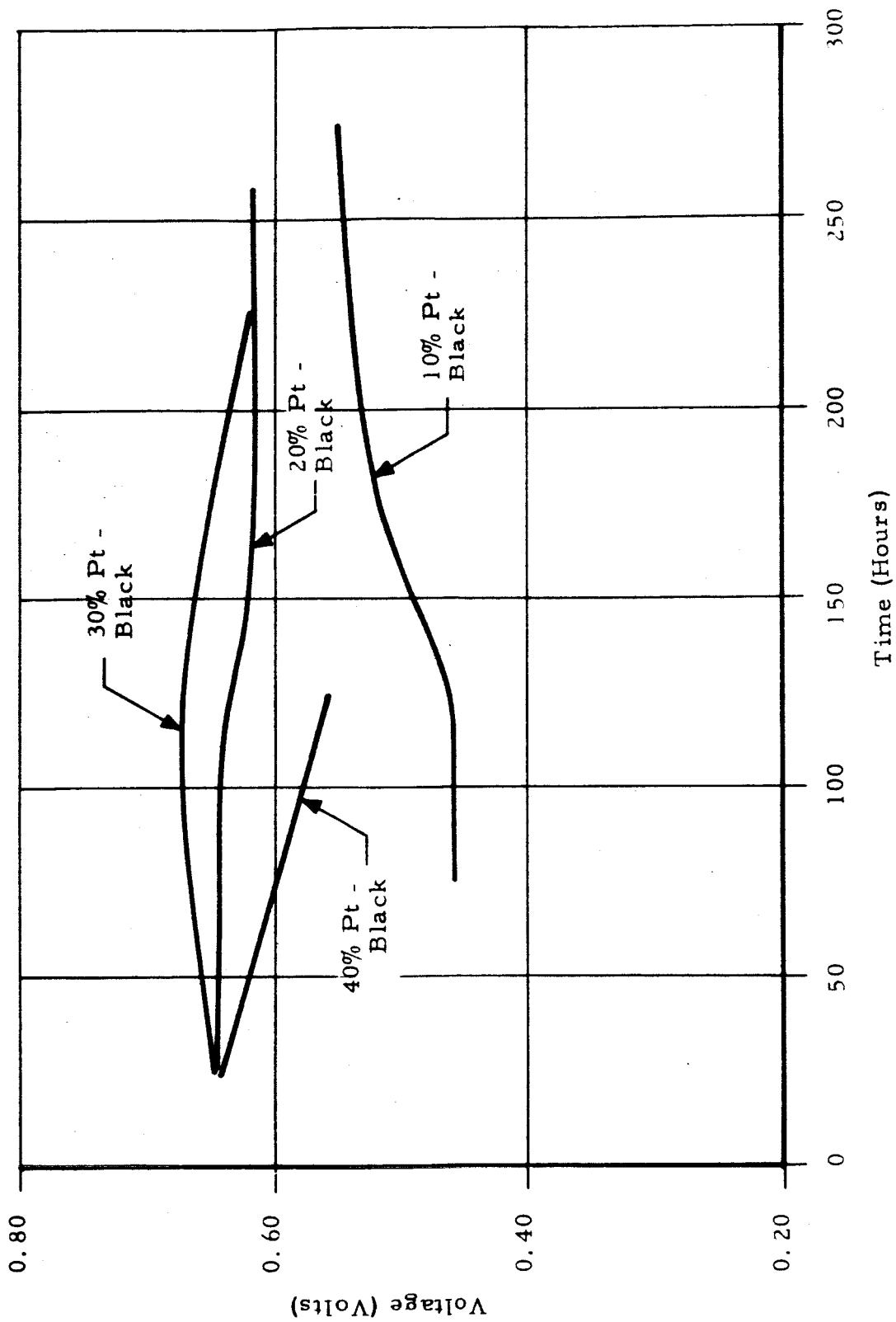
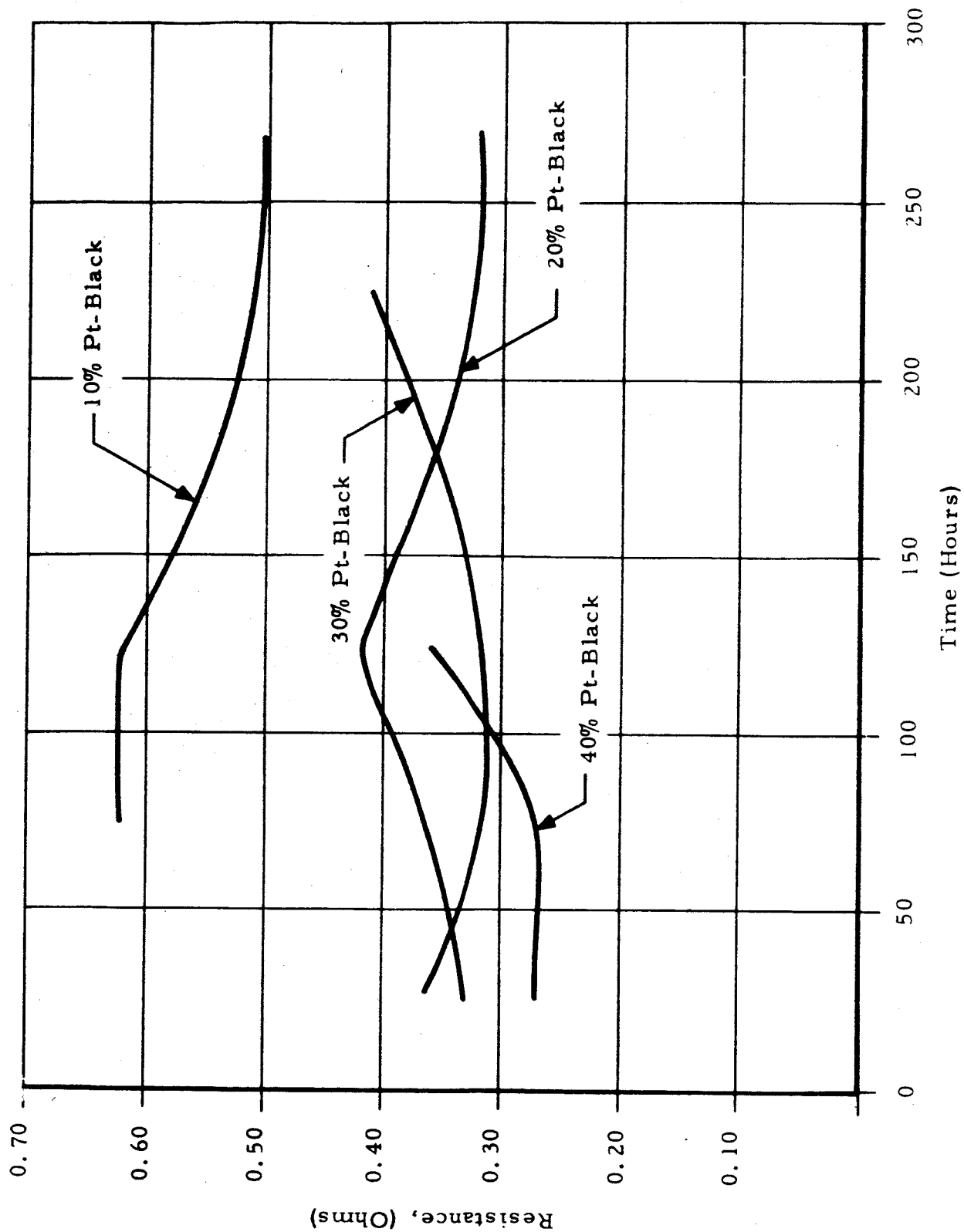


Figure 2. Plot of Fuel Cell Resistances versus Time for Tests at 25°C and at 65°C for Non-Impregnated C200B Membranes at 30 ma/cm²



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Figure 3. Plot of Voltage versus Time for Various Percentages of Platinum Black Impregnated C200B Membranes at $65 \pm 2^\circ\text{C}$ and Current Density of 30 ma/cm^2 . (Add 0.03 volts for leads correction.)



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Figure 4. Plot of Resistances Versus Time for Various Percentages of Platinum Black Impregnated C200B Membrane at $65 \pm 2^\circ\text{C}$

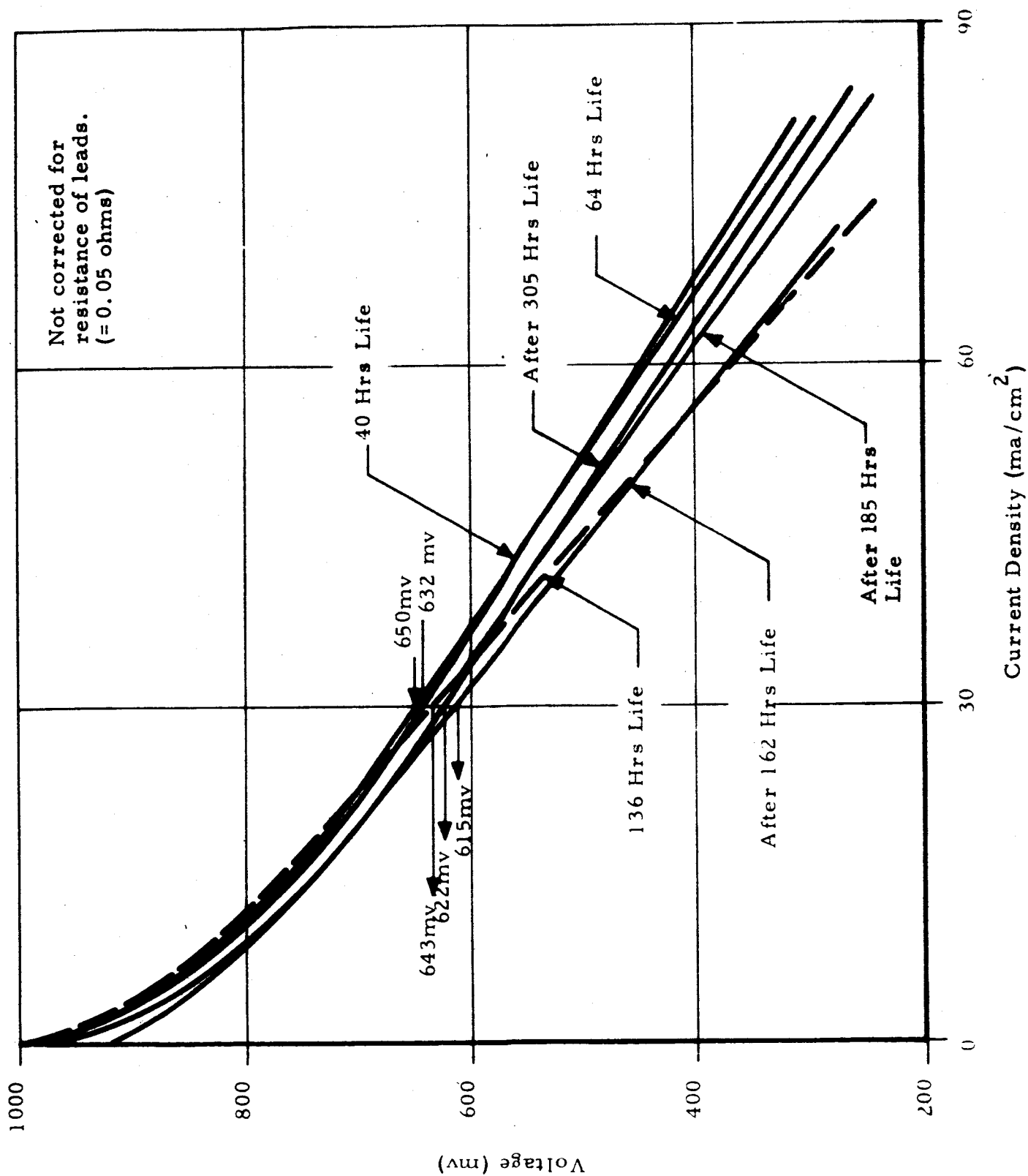


Figure 5. Polarization Curves for Inorganic Membrane Fuel Cell, Test 2, Table I

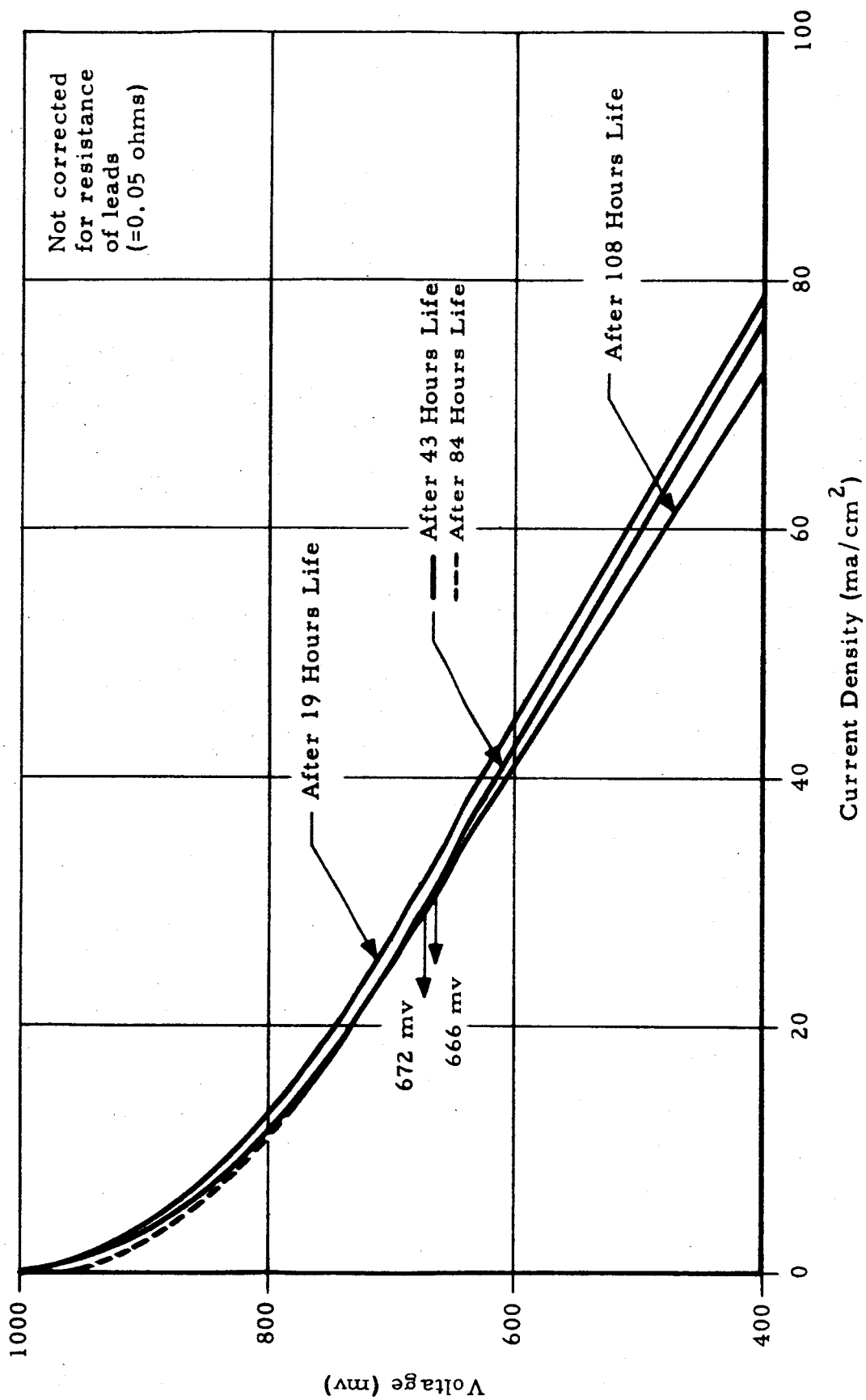


Figure 6. Polarization Curves for Inorganic Membrane Fuel Cells, Test 3, Table I

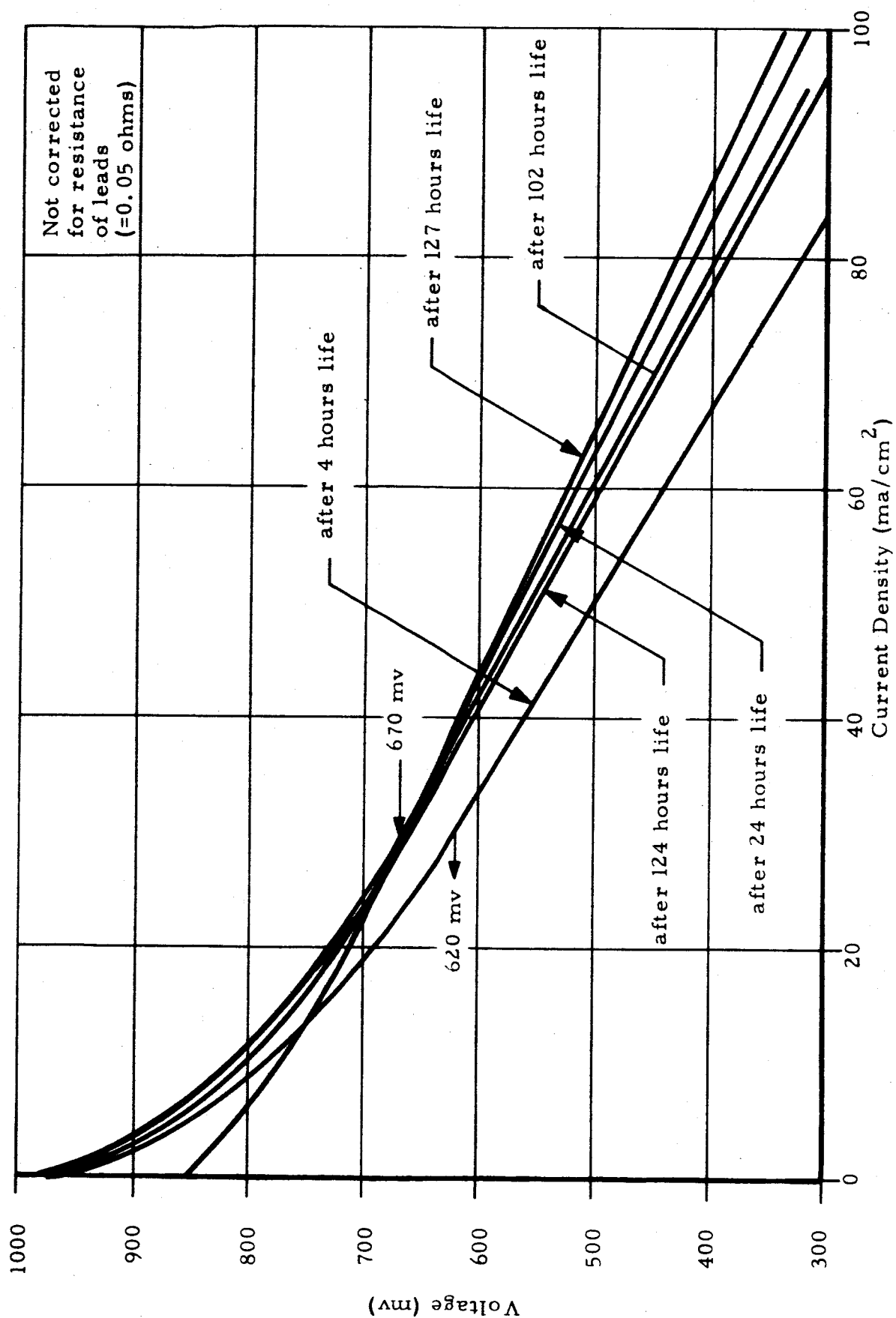


Figure 7: Polarization Curves for Inorganic Membrane Fuel Cell
Test 4 Table I

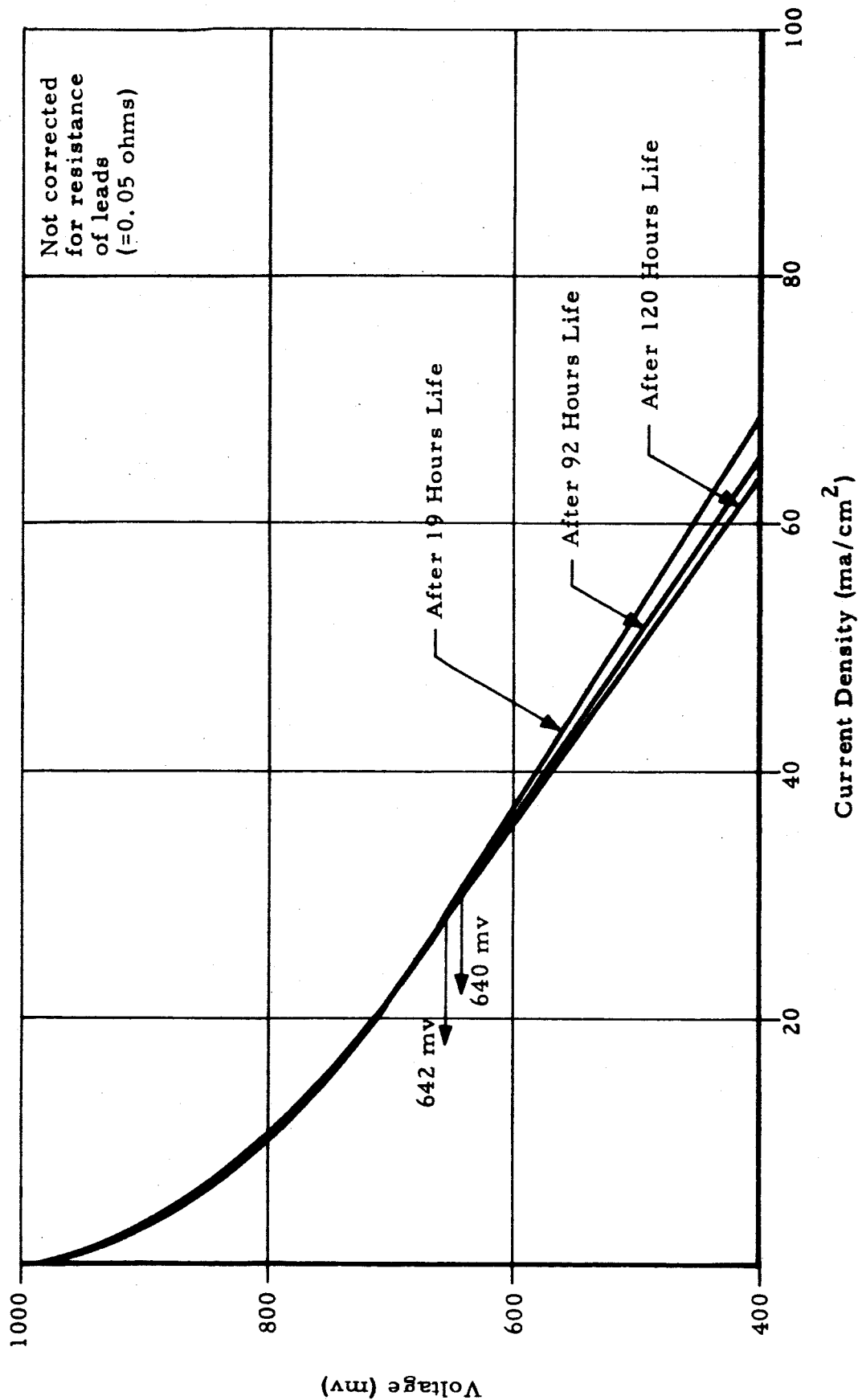


Figure 8. Polarization Curves for Inorganic Membrane Fuel Cells, Test 6, Table I

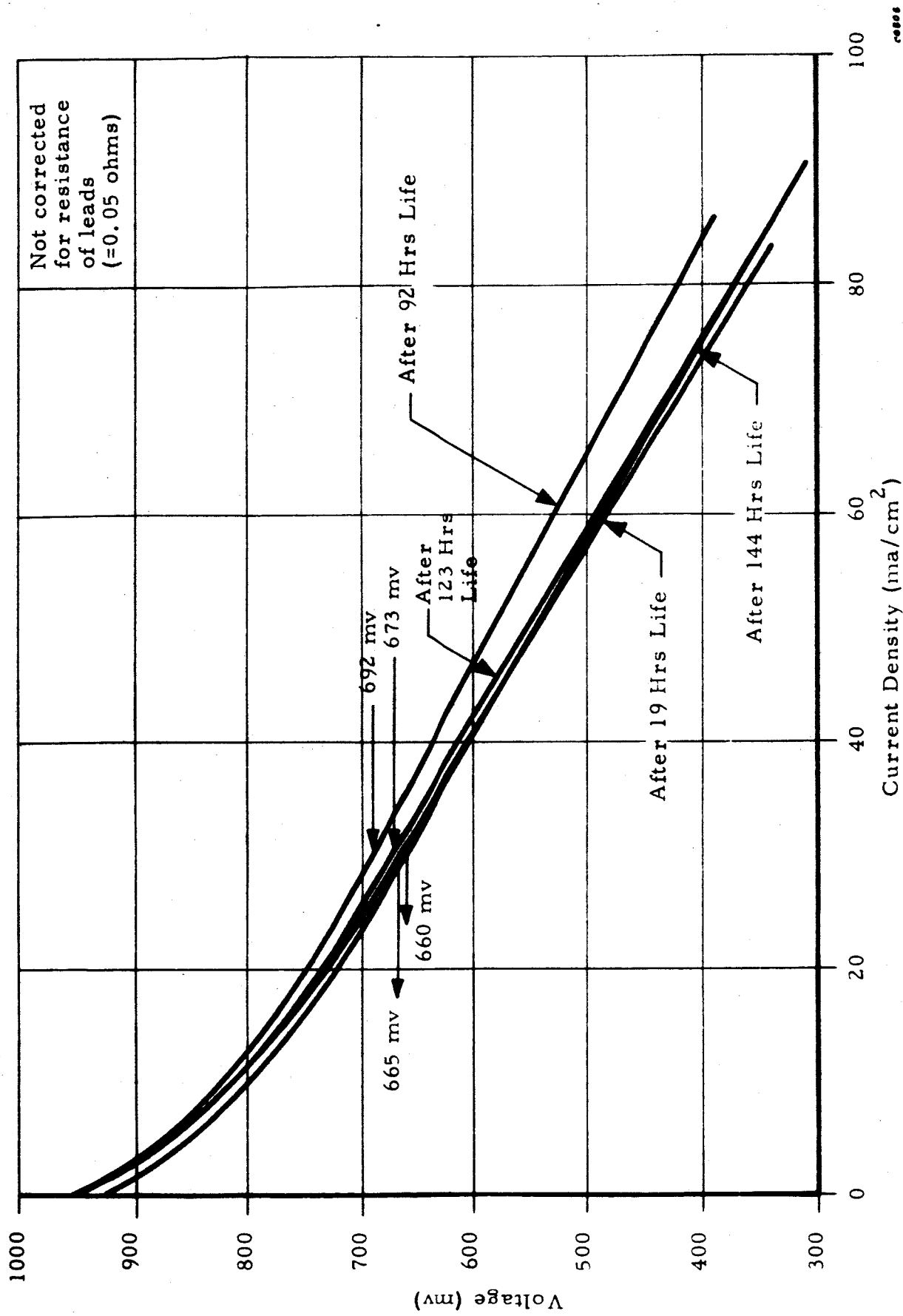


Figure 9. Polarization Curves for Inorganic Membrane Fuel Cell, Test 7, Table I

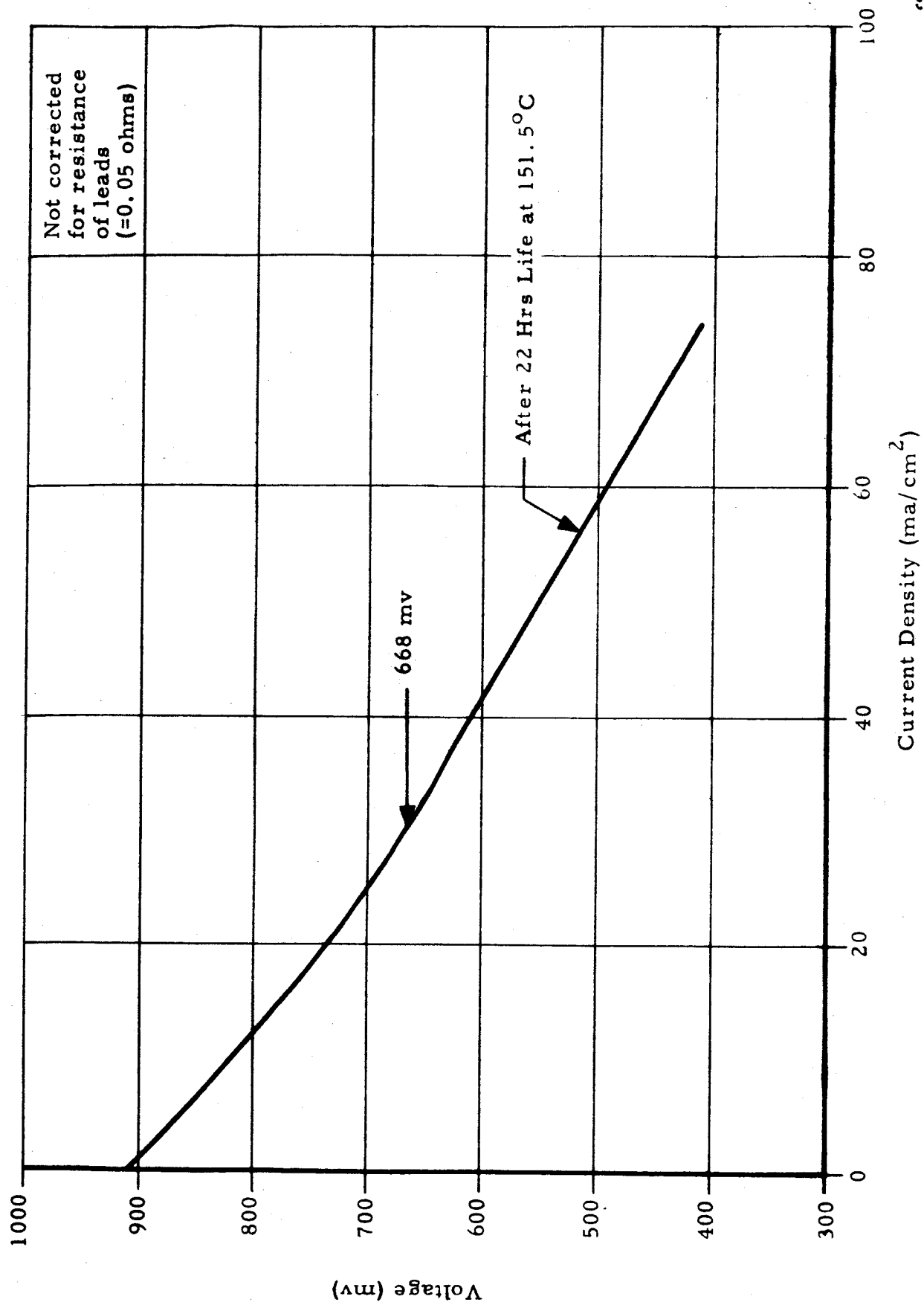
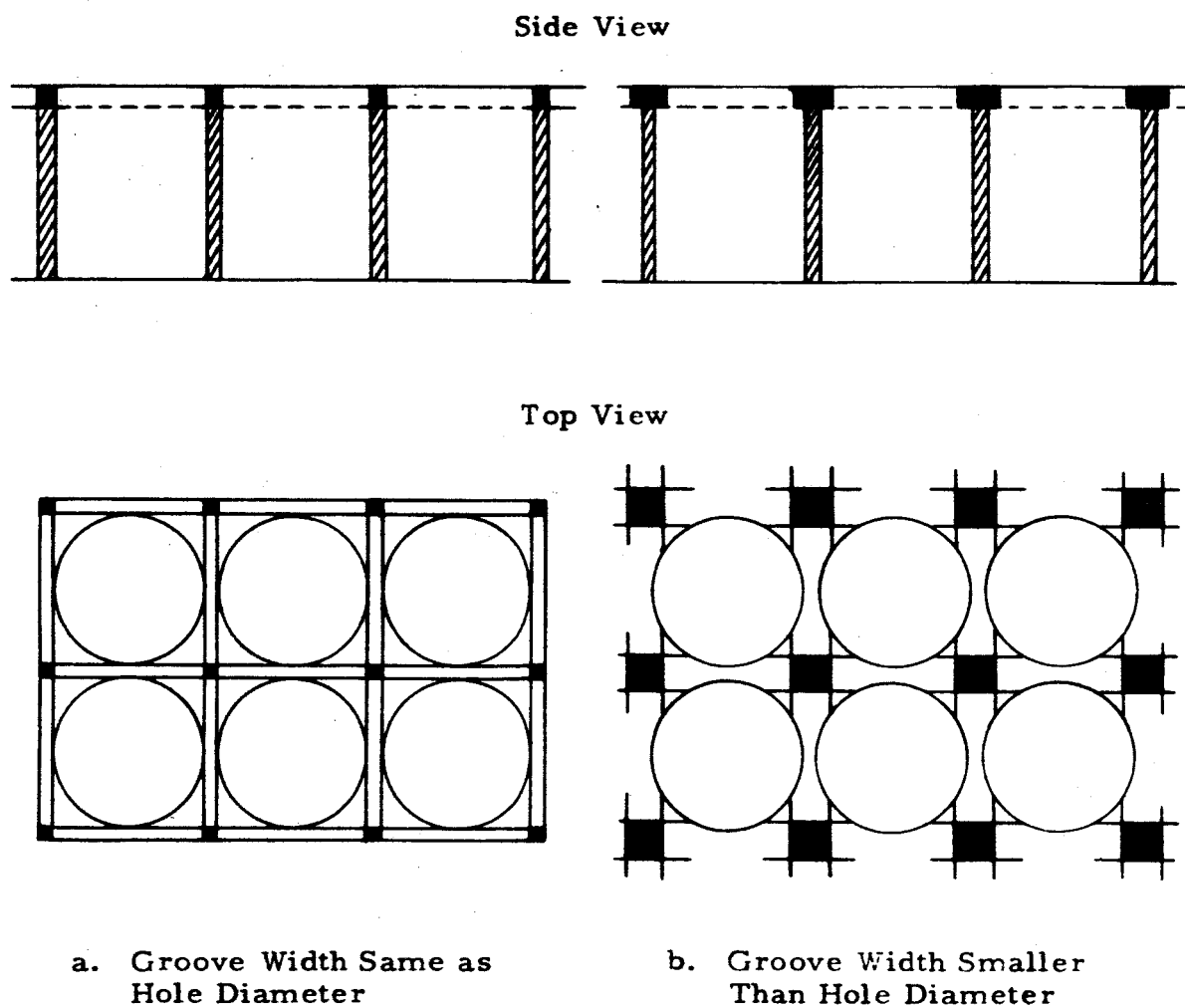
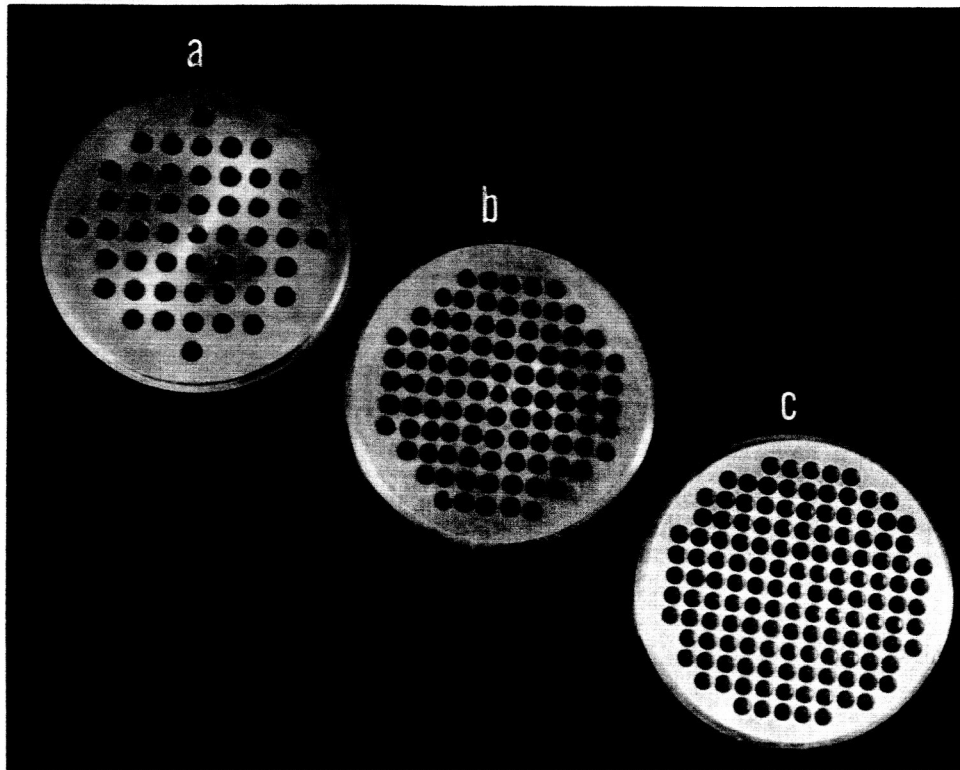


Figure 10. Polarization Curves for Inorganic Membrane Fuel Cell, Test 8, Table I



COJ/J

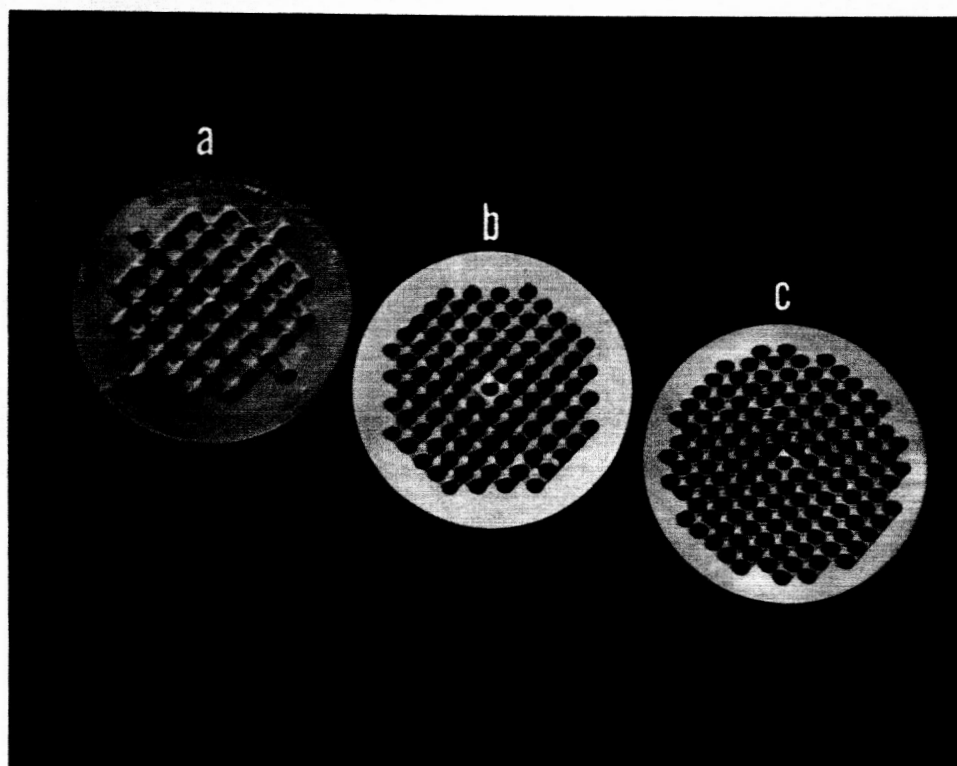
Figure 11. Comparison of Different Layouts of Grooves and Holes for the Electrode Facing Side of the Backup Plates



00831

- (a) Forty-Four holes, 1/8 inch in diameter, in stainless steel backup plate
- (b) Ninety-six holes, 1/8 inch in diameter, in stainless steel backup plate
- (c) One hundred and thirty-six holes, 1/8 inch in diameter, in stainless steel backup plate

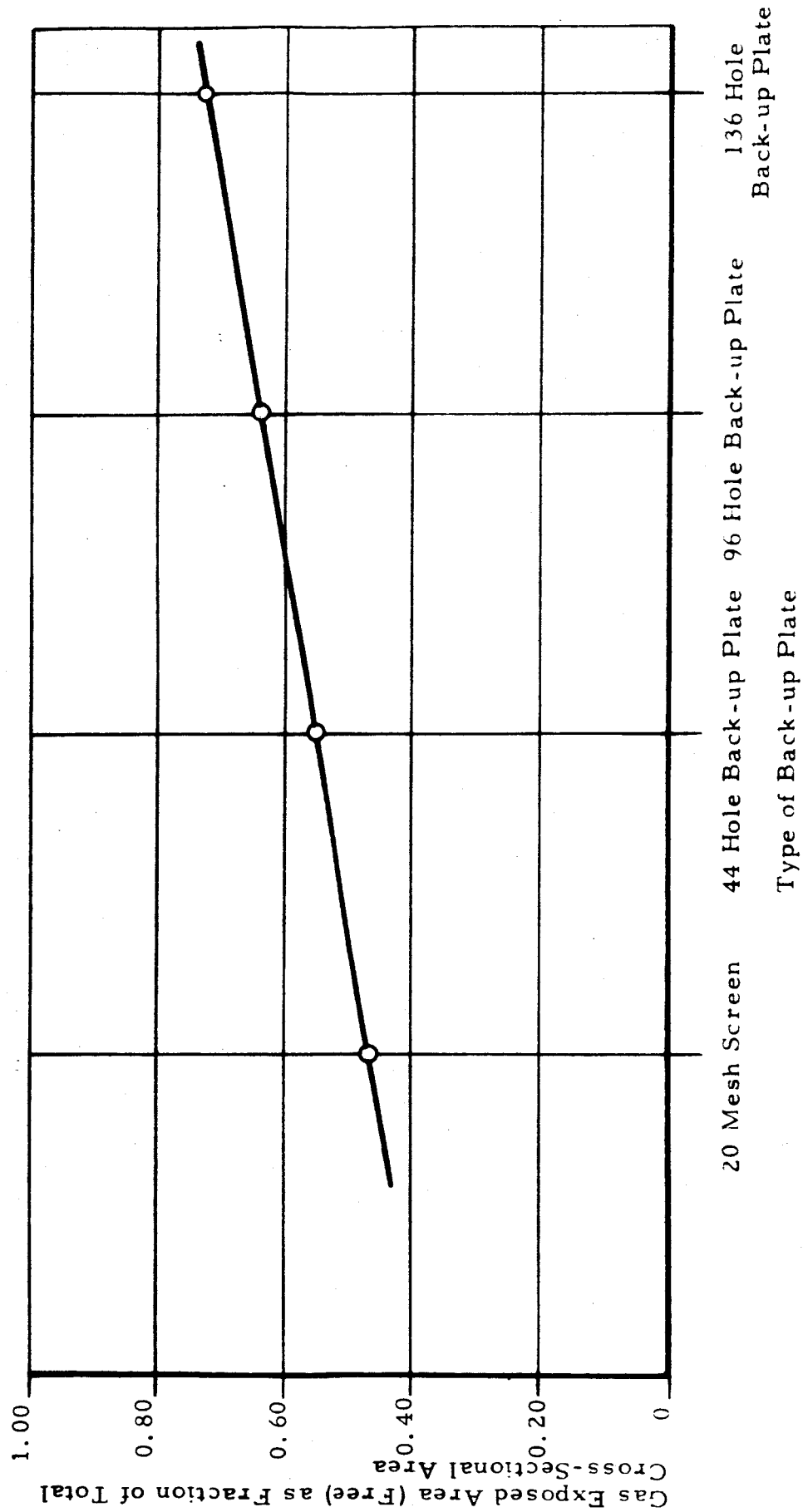
Figure 12. From Left to Right: Three Types of Backup Plates Used in Astropower Compact Fuel Cell (Gas Compartment Face)



C0832

- (a) Forty-four holes, 1/8 inch in diameter, in stainless steel backup plate
- (b) Ninety-six holes, 1/8 inch in diameter, in stainless steel backup plate
- (c) One hundred and thirty-six holes, 1/8 inch in diameter, in stainless steel backup plate

Figure 13. From Left to Right: Three Types of Backup Plates Used in Astropower Compact Fuel Cell (Electrode Face)



10077

Figure 14. Relationship of Areas of Four Different Types of Back-up Plates Used in the Compact Fuel Cell Design

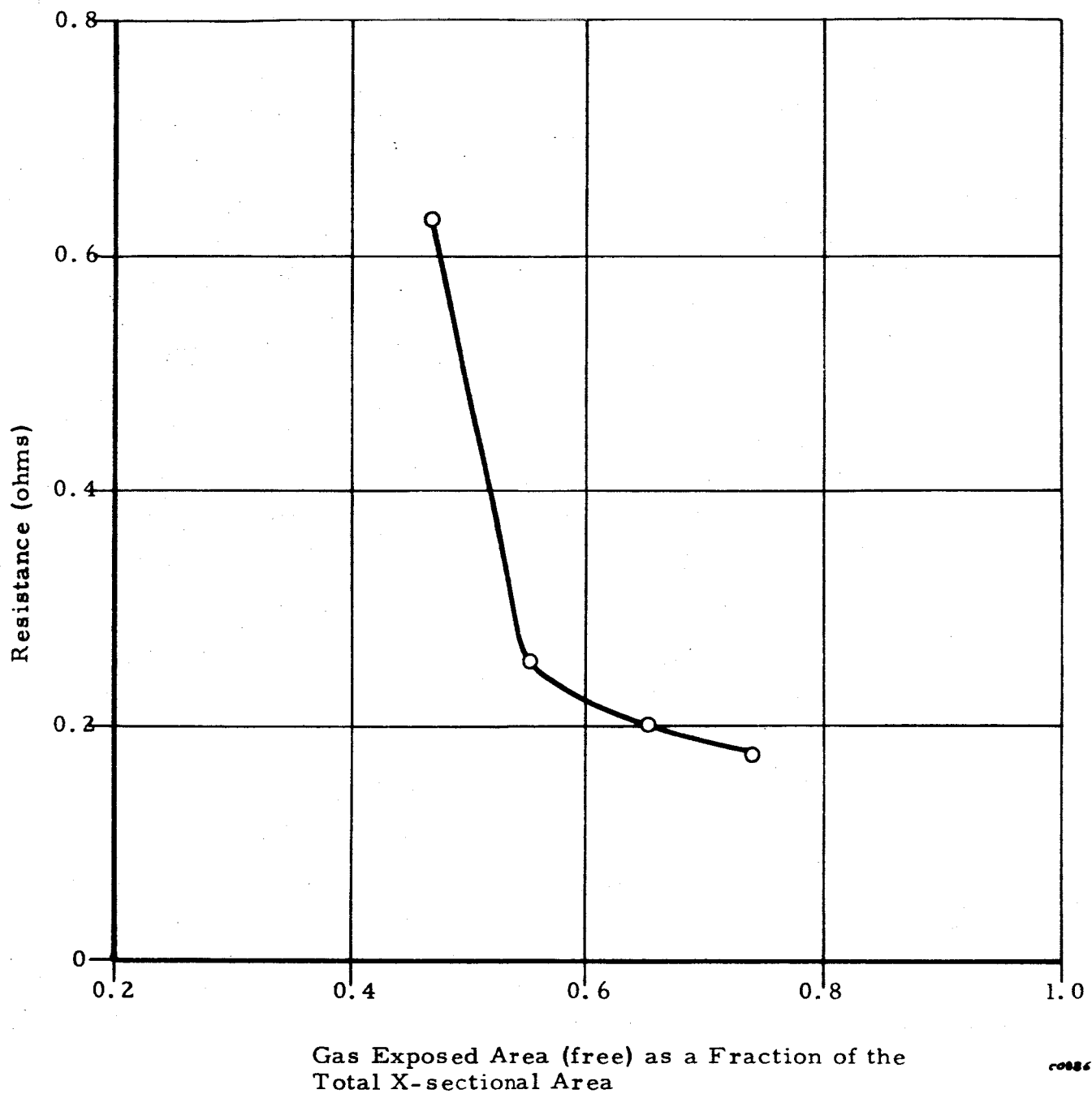
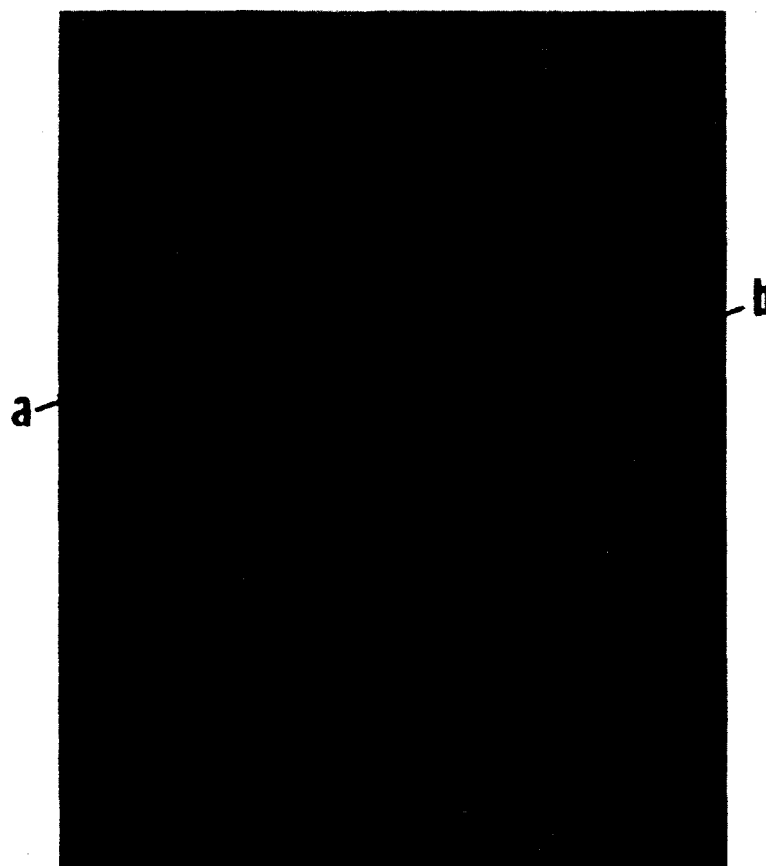


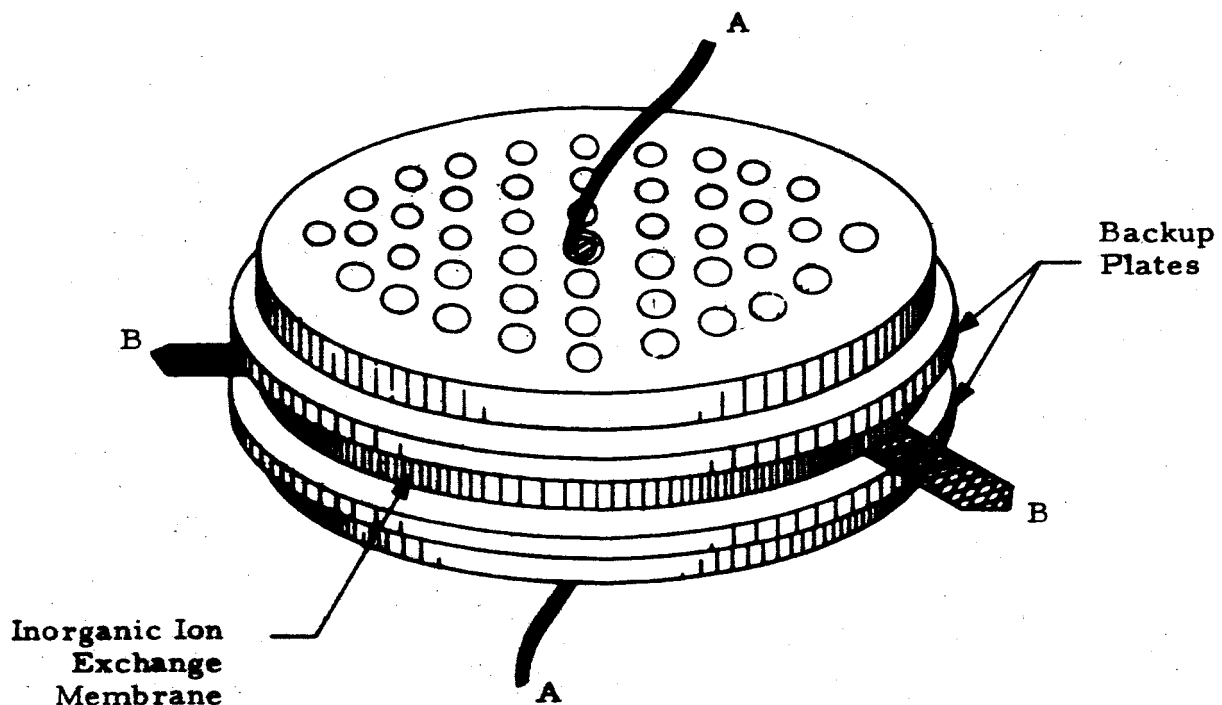
Figure 15. Relationship of Fuel Cell Resistance with Available Electrode Area Exposed to the Reactant Gases



60889

- (a) dark area — exposed to gas
- (b) light grey area — contact points for electrical lead off and to maintain equal pressure against the electrode-membrane composite

Figure 16. Two-Inch Diameter American Cyanamid AA-1 Electrode and Platinum Black Deposit, Showing the Contact Points, Resulting from the Lugs of the Back-up Plate

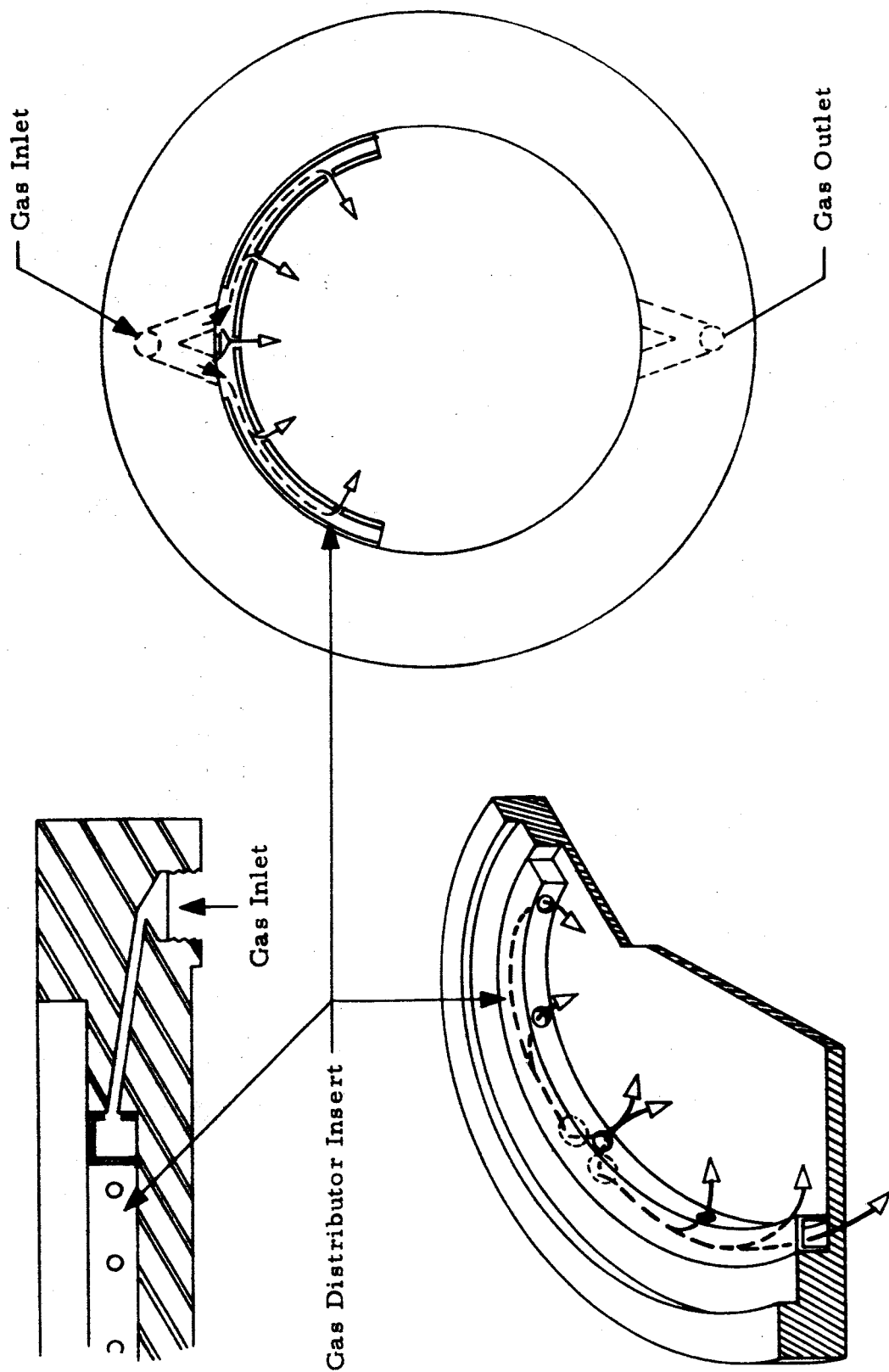


Electrical Leads Connected to

A-A - Backup Plates

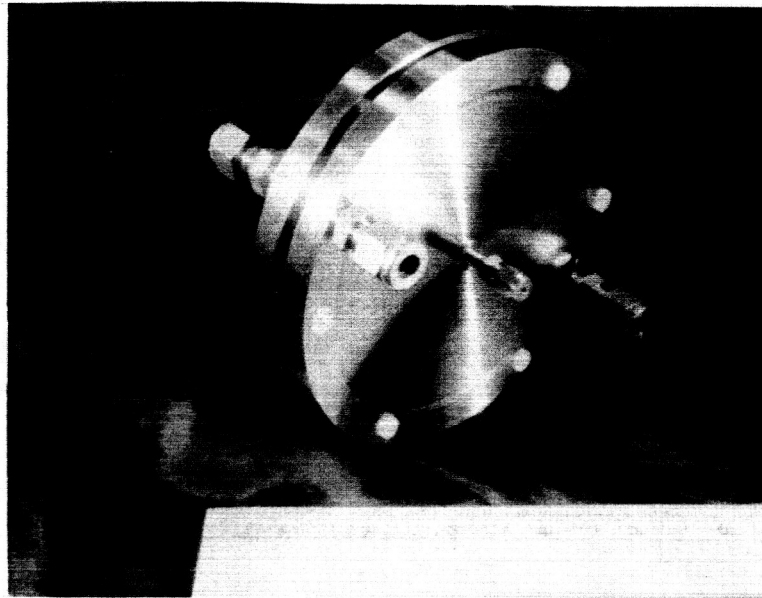
C C B-B - American Cyanamid Type A A-1 Electrode Tabs

Figure 17: Inorganic Ion Exchange Membrane Backup Plate Sandwich, Illustrating Electrical Lead Off Via (a) Backup Plates (b) Electrode Tabs.

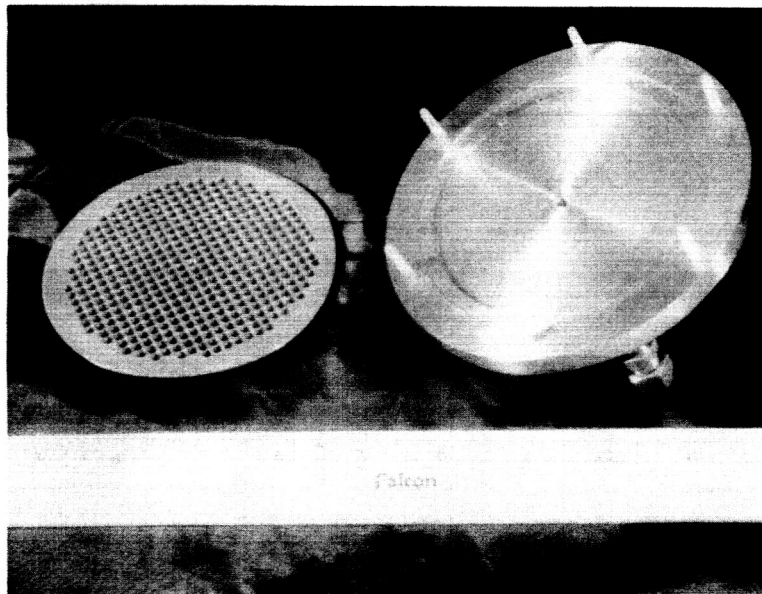


00012

Figure 18. Large Compact Fuel Cell and Gas Distribution Insert



C0883



C0884

Figure 19. Large Astropower Compact Fuel Cell

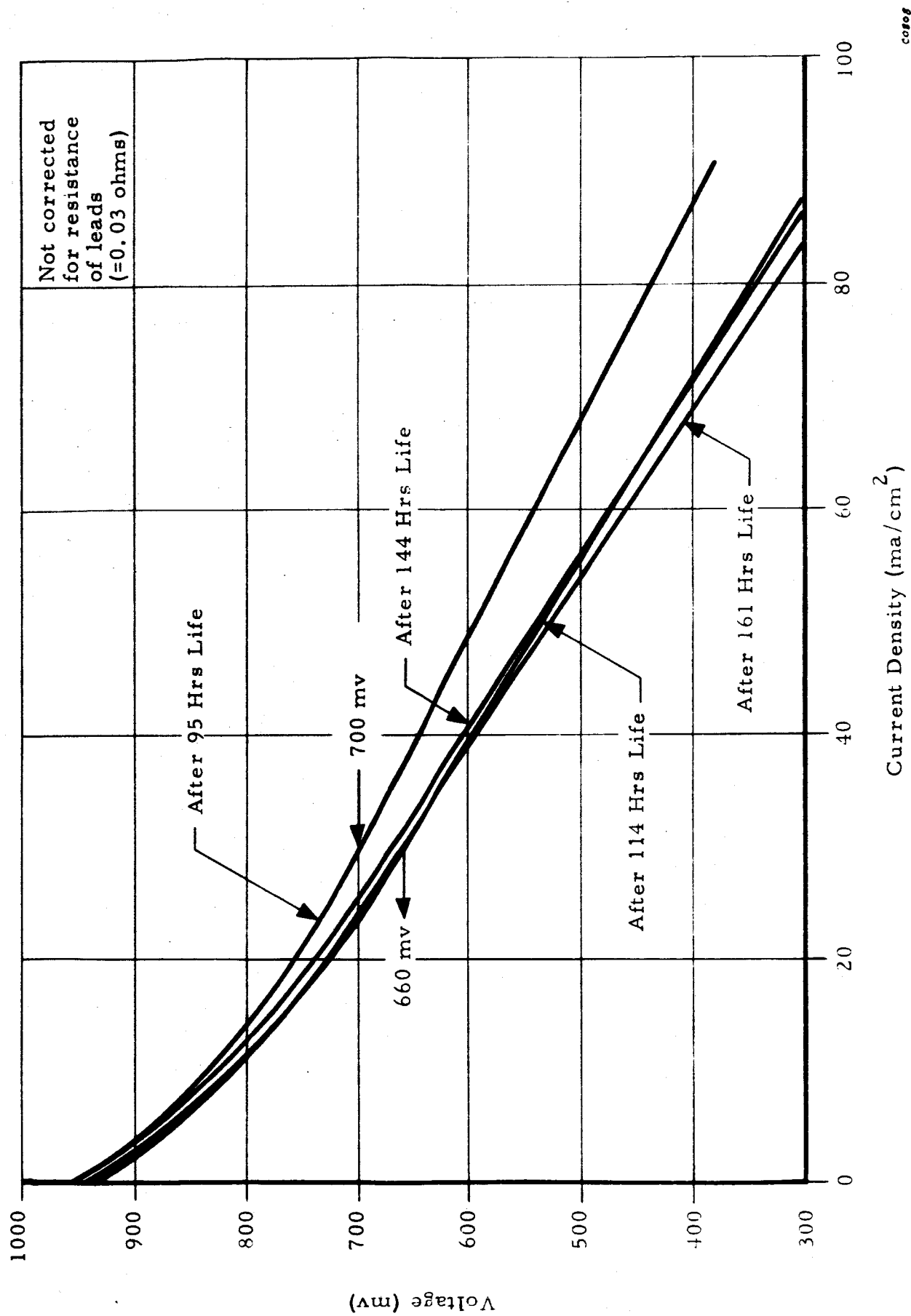


Figure 20. Polarization Curves for Inorganic Membrane Fuel Cell
Test 9, Table II

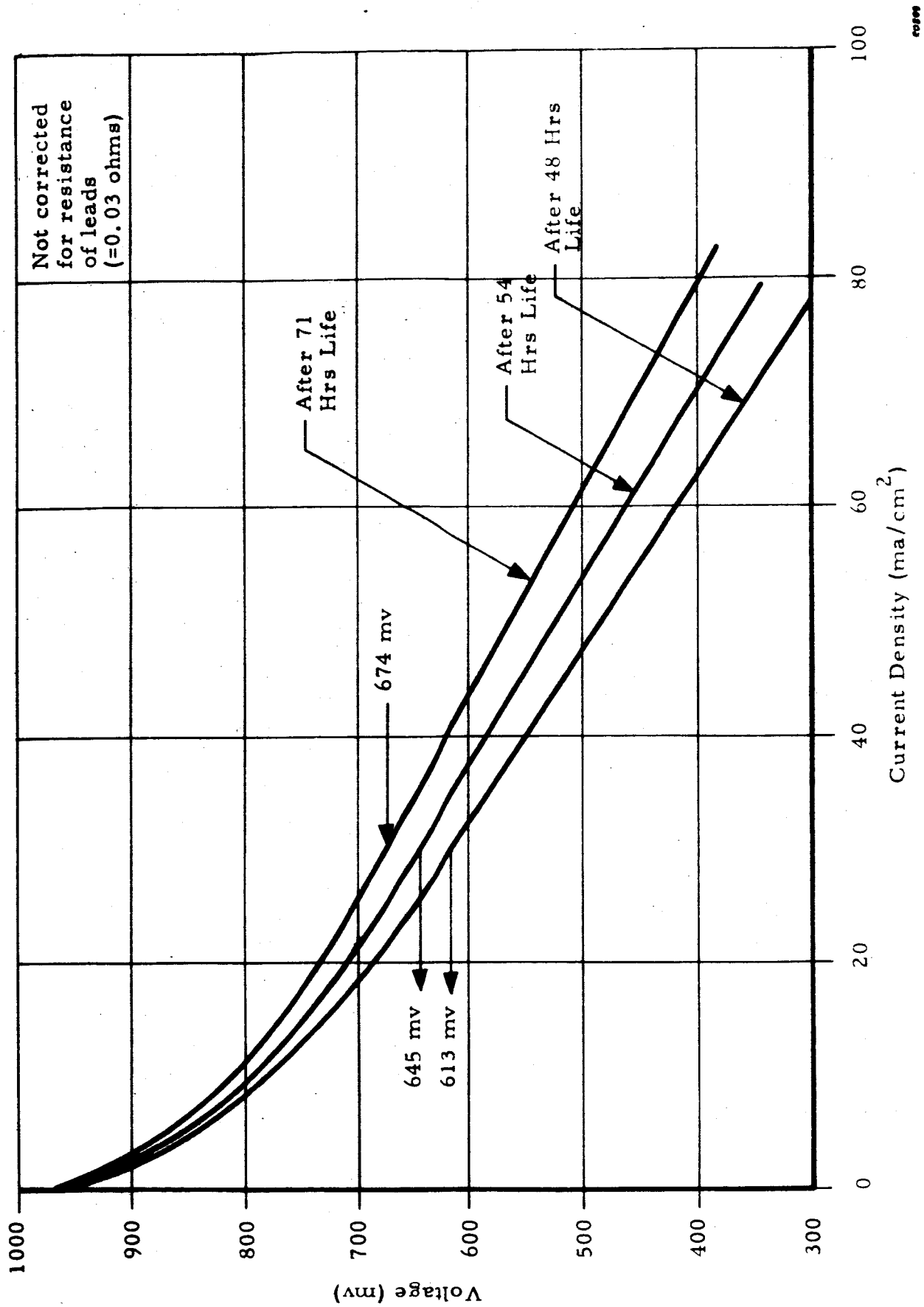


Figure 21. Polarization Curves for Inorganic Membrane Fuel Cell
Test 10, Table II

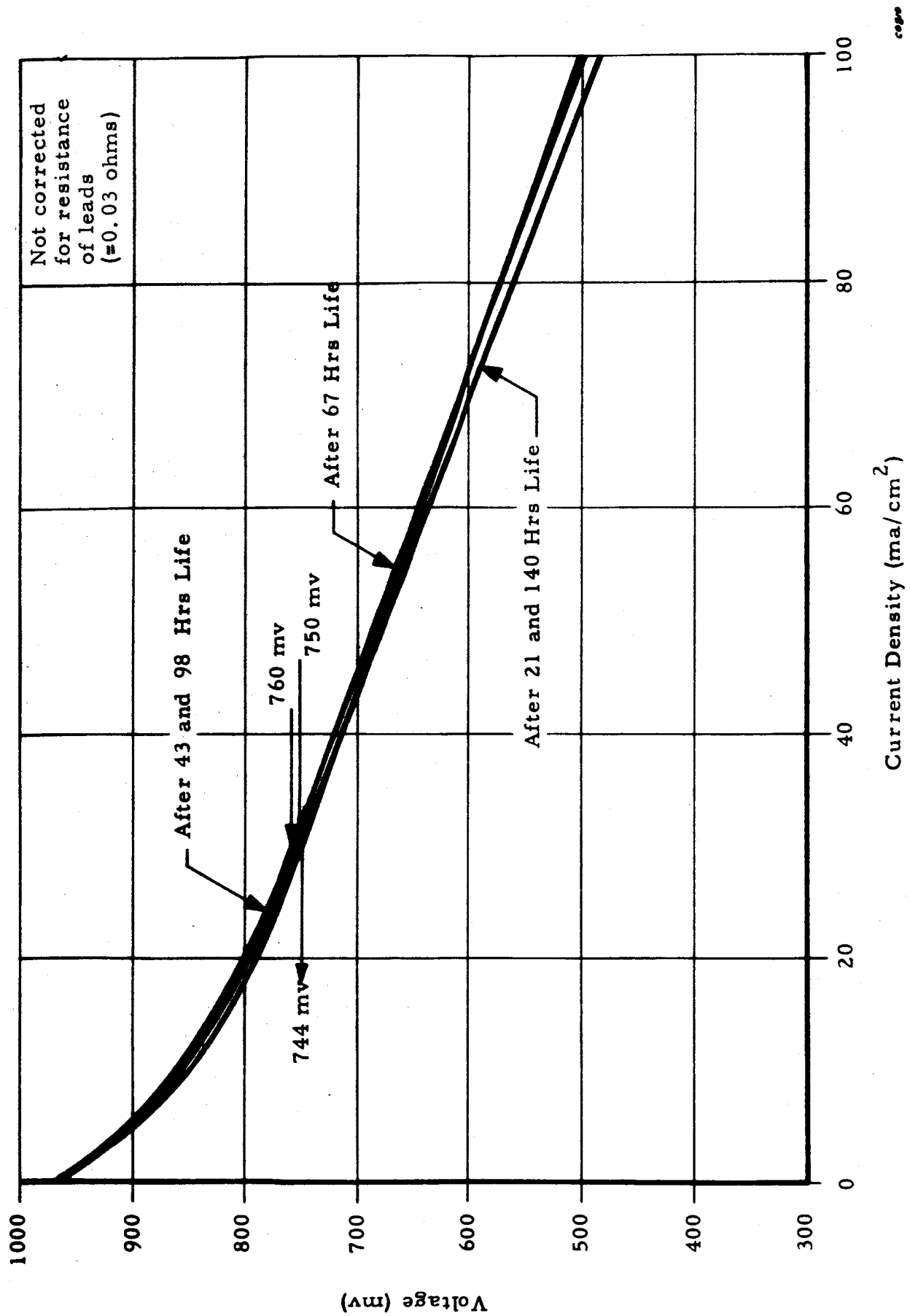


Figure 22. Polarization Curves for Inorganic Membrane Fuel Cell
Test II, Table II

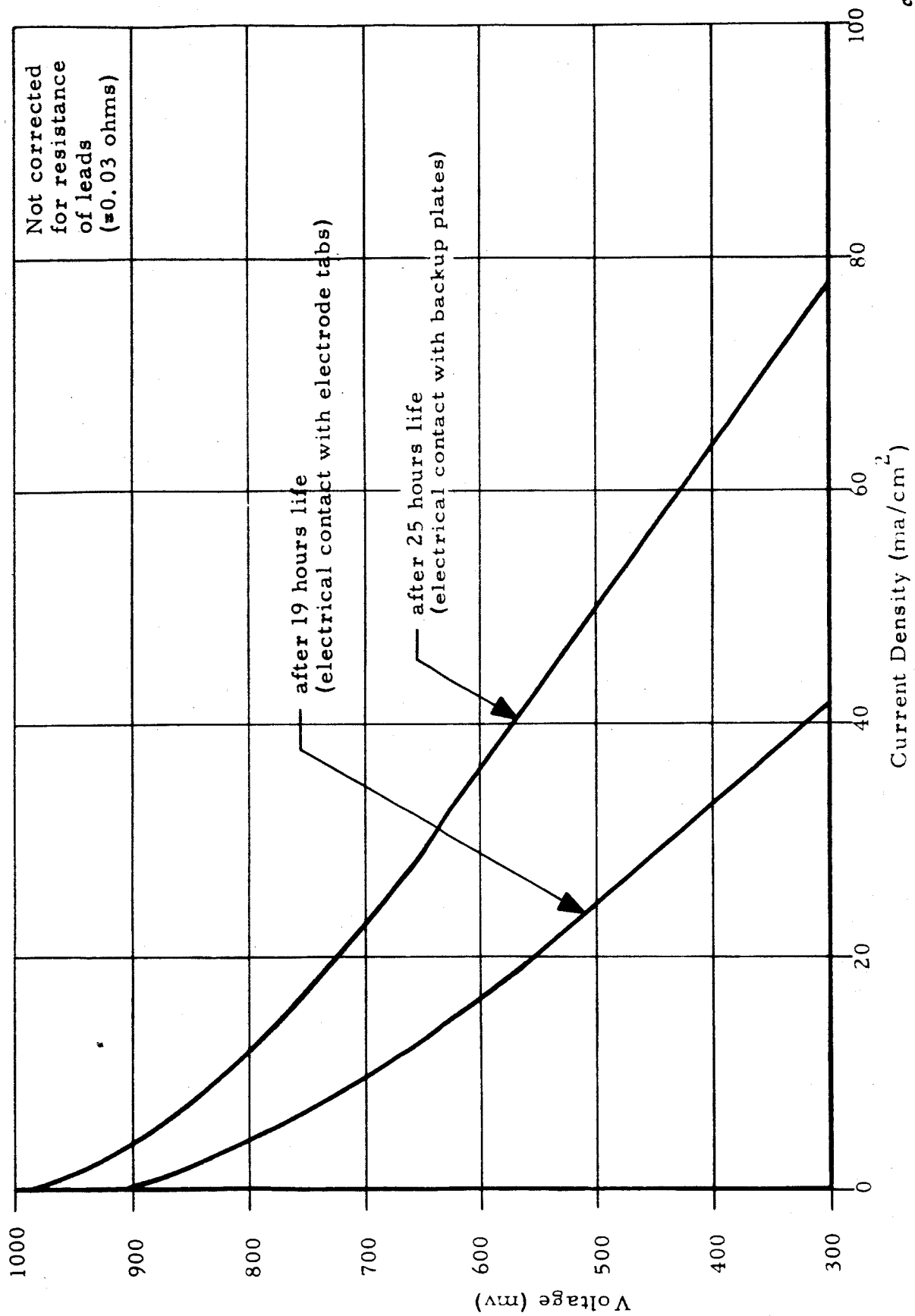


Figure 23. Polarization Curves for Inorganic Membrane Fuel Cell
Test 12 Table II

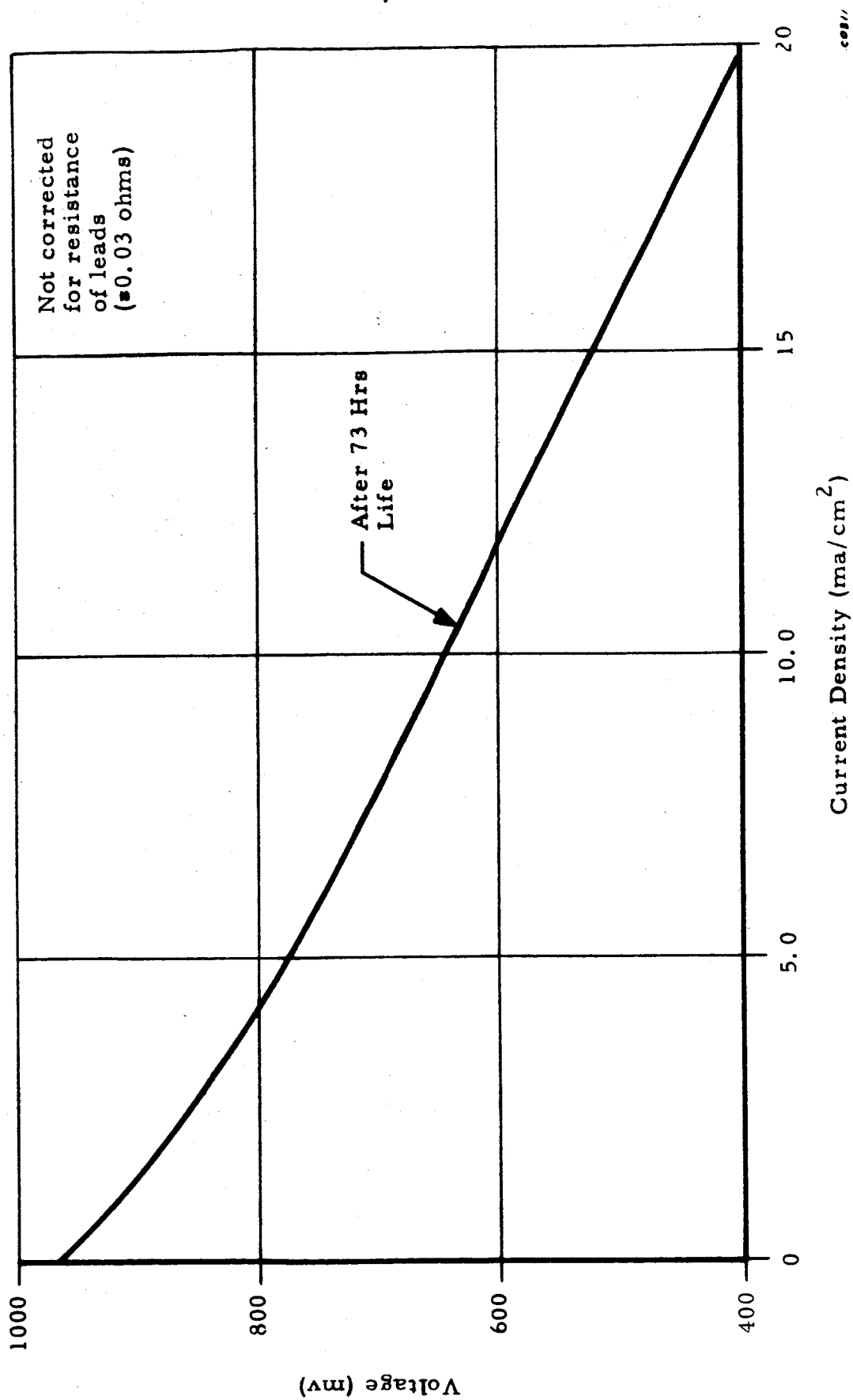


Figure 24. Polarization Curve for Inorganic Membrane Fuel Cell
Test 14, Table II

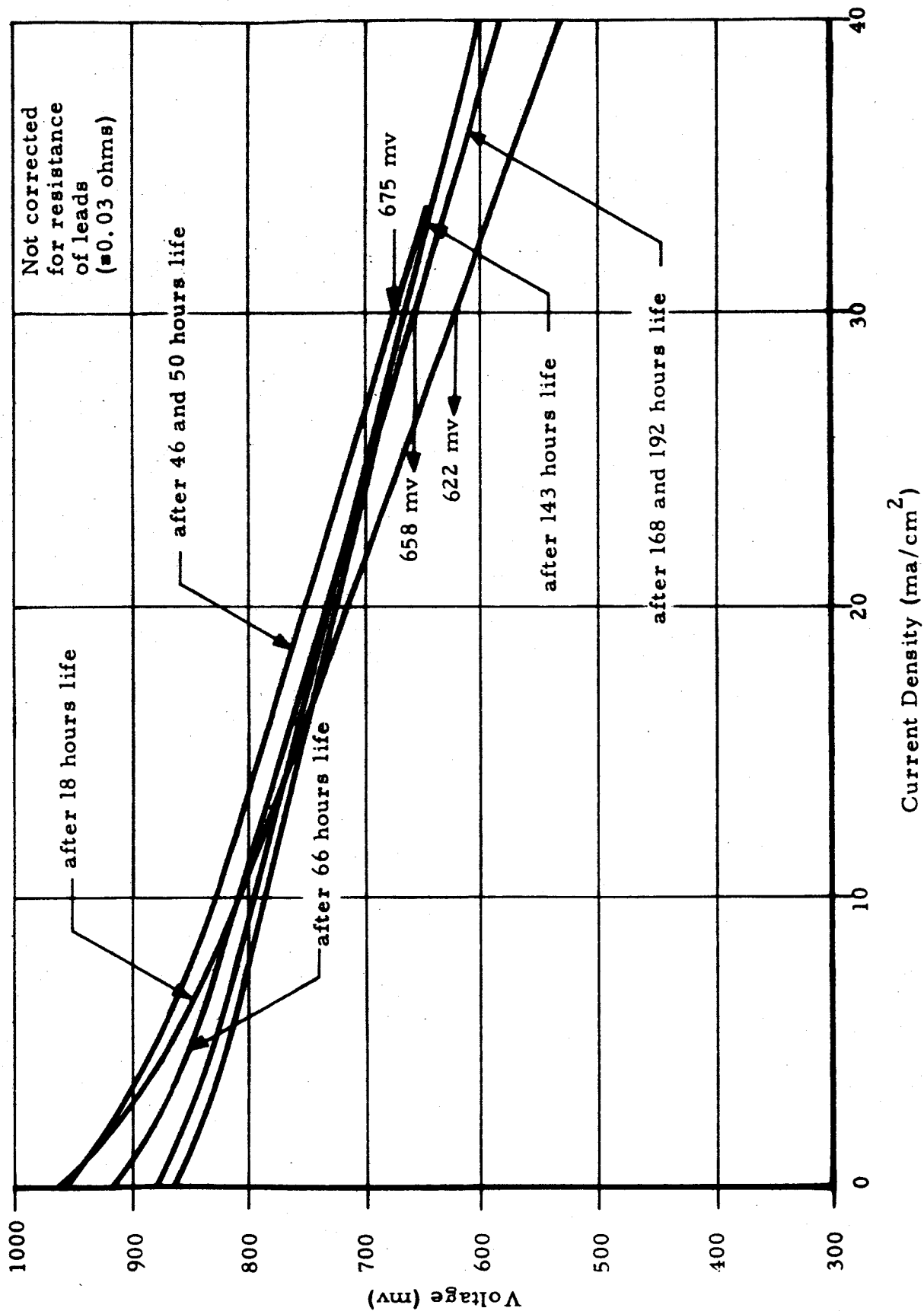
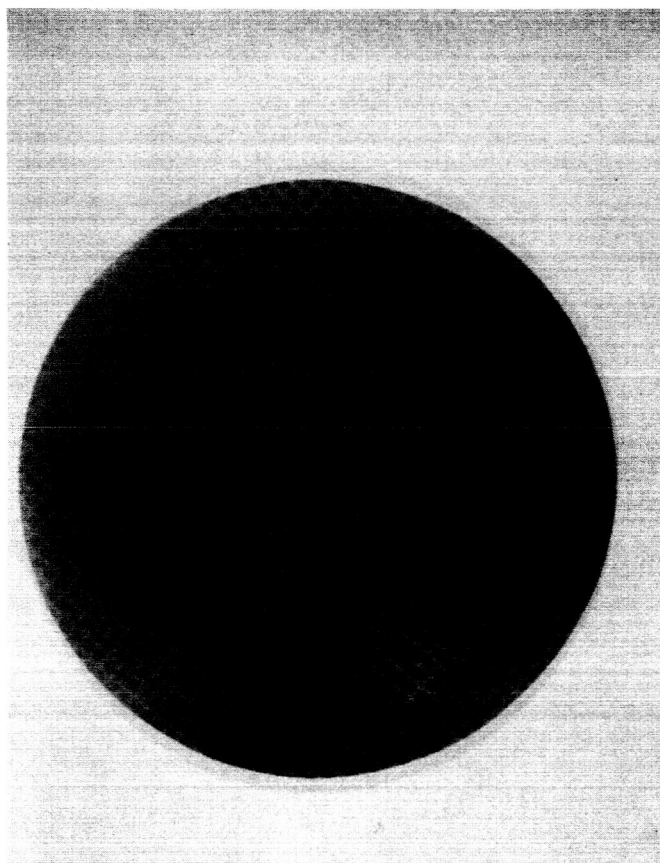
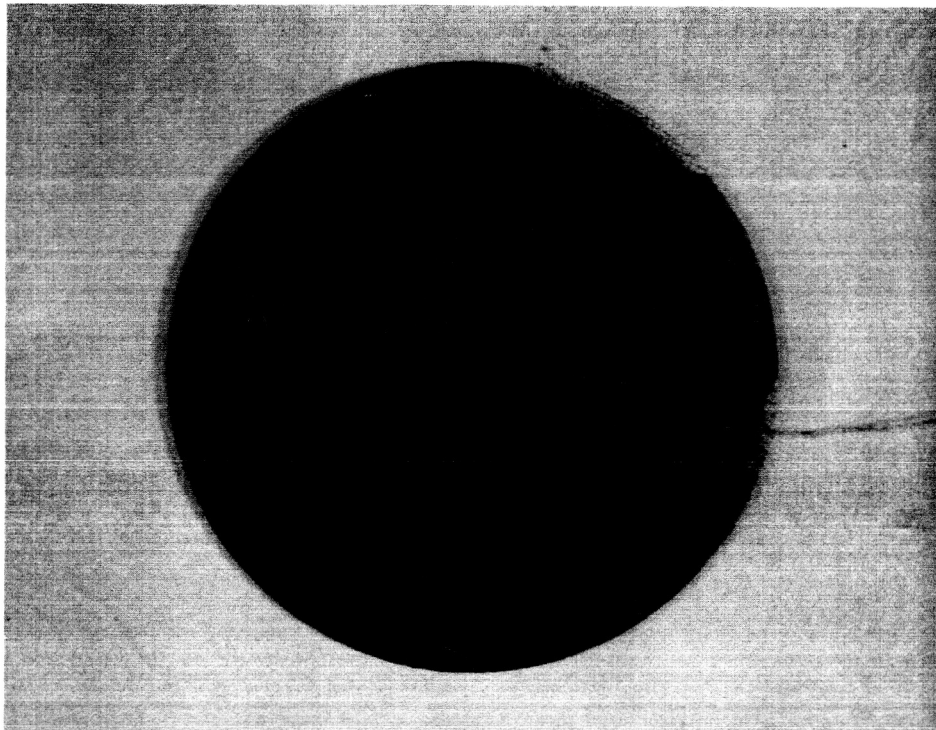


Figure 25. Polarization Curves for Inorganic Membrane Fuel Cell
Test 15, Table II



ca885

Figure 26. Photograph of Unitized Electrode-Catalyst-Membrane Configuration (Front View)



00030

Figure 27. Unitized, Fused C200B Membrane-Electrode Configuration, Demonstrating Silver Wire Leads Interwoven Through Electrode Screen Structure

TABLE I

**SUMMARY OF SIGNIFICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE -
PHOSPHORIC ACID - "ZEOLON-H" C200B MEMBRANE PERFORMED IN THE
ASTROPOWER ANALYTICAL FUEL CELL DESIGN(a)**

Fuel Cell Test No.	Membrane Description	Membrane Thickness, mm	Temperature, °C	Current Density at 0.5 volts, ma/cm ²	Voltage (f) at 30 ma/cm ² , volts	Fuel Cell Resistance, ohms	Open Circuit Voltage, volts	Time of Measurement from Start, hours	Life Time of Test(b) hours
1	No special treatment	0.51	25 ± 1	27.5 26.5 26.0 26.0 25.5 24.8 25.5	0.464 0.453 0.444 0.444 0.435 0.420 0.435	0.66 0.66 0.69 0.69 0.73 0.75 0.73	0.953 0.962 0.967 0.983 0.982 0.975 0.982	98 140 166 231 255 279 340	340(c-1)
2	Impregnated with platinum black - 20% in both outer one-third layers	1.07	65 ± 2	52.2 51.8 45.0 44.2 48.5 50.2 49.5 48.2	0.643 0.650 0.632 0.615 0.622 0.632 0.628 0.632	0.32 0.34 0.43 0.40 0.32 0.32 0.32 0.35	0.980 0.985 0.970 0.925 0.960 0.985 0.968 0.977	40 64 136 162 185 212 233 305	305(c-1)
3	Impregnated with platinum black - 20%(e) in both outer one-twentieth layers	0.91	65 ± 2	61.8 59.5 59.5 56.5 -	0.690 0.672 0.672 0.666 0.545	0.30 0.29 0.29 0.32 -	0.985 0.960 0.987 0.950 -	19 43 84 108 246	250(c-1)
4	Impregnated with 2% Teflon and platinum black - 20% in both outer one- twentieth layers and then fired at 350°C	0.89	65 ± 2	50.0 63.5 60.5 59.5 65.0	0.620 0.670 0.664 0.663 0.662	0.29 0.26 0.26 0.28 0.24	0.958 0.975 0.980 0.985 0.851	4 24 102 124 127	188(c-1)
5	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.74	65 ± 2	75.0	0.700	0.22	0.985	19	90(c-3)
6	Impregnated with platinum black - 20%(d) in both outer one-third layers	0.69	65 ± 2 102 ± 2 65 ± 1	52.0 50.0 50.0	0.642 0.640 0.640	0.31 0.35 0.34	0.983 1.000 0.980	19 92 120	123(c-1)
7	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.87	70 ± 1	65.7 57.0 59.0 57.7	0.673 0.692 0.665 0.660	0.32 0.27 0.28 0.29	0.953 0.960 0.965 0.930	19 92 123 144	144(c-2)

TABLE I (Continued)

**SUMMARY OF SIGNIFICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE -
PHOSPHORIC ACID - "ZEOLON-H" C200B MEMBRANE PERFORMED IN THE
ASTROPOWER ANALYTICAL FUEL CELL DESIGN(a)**

Fuel Cell Test No.	Membrane Description	Membrane Thickness mm	Membrane Temperature, °C	Current Density at 0.5 volts, ma/cm ²	Voltage at 30 ma/cm ² , volts	Fuel Cell Resistance, ohms	Open Circuit Voltage, volts	Time of Measurement from Start, hours	Life Time of Test(b) hours
8	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.85	151 ± 1	58.5	0.668	0.29	0.917	22	40(c-1)

(a) All tests were run at constant current densities of 30 ma/cm² with membranes having cross-sectional area of 20.2 cm².

(b) Tests run for less than 300 hours were terminated because of diminishing fuel cell performance.

(c) Tests were run with (1) 44-hole back-up plate and 0.125 inch groove width
(2) 96-hole back-up plate and 0.125 inch groove width
(3) 136-hole back-up plate and 0.105 inch groove width.

(d) Membrane surface was Teflon sprayed and then resintered at 300°C.

(e) Membrane prepared from hydrogen-exchanged "Zeolon-H".

(f) These values are uncorrected for IR drop through leads from the test cell to the voltmeter. The typical correction would be 0.03 volts added to each voltage reading in this column.

TABLE II

SUMMARY OF SIGNIFICANT FUEL CELL TESTS ON PRESINTERED ZIRCONIUM DIOXIDE -
PHOSPHORIC ACID - "ZEOLON-H" C200B MEMBRANE TESTS PERFORMED IN
THE ASTROPOWER COMPACT FUEL CELL DESIGN (a)

Fuel Cell Test No.	Membrane Description	Membrane Thickness, mm	Temperature, °C	Current Density at 0.5 volts, ma/cm ²	Voltage (e) at 30 ma/cm ² , volts	Fuel Cell Resistance, ohms	Open Circuit Voltage, volts	Time of Measurement from Start of Run, hours	Time of Run, (b) hours
9	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.86	65 ± 3	68.0 55.8 56.0 54.0	0.700 0.660 0.670 0.660	0.27 0.31 0.32 0.33	0.972 0.950 0.952 0.935	95 114 144 161	161 (c-1)
10	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.75	65 ± 1	47.5 53.5 61.5	0.613 0.645 0.674	0.32 0.31 0.27	0.955 0.960 0.962	48 54 71	75 (c-2)
11	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.70	65 ± 2	95.6 99.0 99.7 99.0 95.6 95.6	0.750 0.760 0.750 0.760 0.750 0.750	0.20 0.19 0.18 0.19 0.20 0.20	0.965 0.975 0.970 0.965 0.962 0.960	21 43 67 98 140 164	243 (c-3)
12	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.95	65 ± 2	24.5 50.5	0.436 0.643	0.57 0.35	0.915 0.988	19 25	45 (c-2), (d-1), (c-2), (d-2)
13	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.75	65 ± 2	72.5	0.710	0.24	0.980	28	104 (c-3)
14	No special treatment	1.04	65 ± 1	15.9	-	0.31	0.965	73	74 (c-4)
15	Impregnated with platinum black - 20% in both outer one-twentieth layers	0.95	65 ± 2	43.5 51.2 51.2 51.2 56.2 51.2 51.2	0.622 (f) 0.675 0.675 0.658 0.667 0.667 0.658	0.11 0.09 0.09 0.09 0.08 0.08 0.08	0.965 0.960 0.958 0.910 0.880 0.860 0.858	18 46 50 66 143 168 192	216 (c-4)

(a) All tests were run at constant current densities of 30 ma/cm² with membranes having cross-sectional areas of 20.2 cm², except tests No. 14 and 15 which had cross sectional areas of 80.8 cm² and diameters of 4 inches.

(b) Tests were terminated at indicated number of hours because of diminished performance. (c) Add 0.02 volts for leads correction except for Test 15.

(c) Tests were run with (1) 96-hole and 0.125 inch groove width gold plated back-up plate (f) Add 0.08 volts for leads correction.

(2) 136-hole and 0.125 inch groove width back-up plate

(3) 136-hole and 0.105 inch groove width back-up plate

(4) 360-hole and 0.125 inch groove width back-up plate (4 inch diameter)

(d) Polarization terminal leads were connected to (1) electrode tabs on either side of membrane

(2) regular back-up plate screws.

TABLE III

SUMMARY OF RESULTS OF ANALYTICAL FUEL CELL TESTS WITH
PALLADIUM AND IRIUM CATALYST MATERIALS

Fuel Cell Test No.	Electrode Description (a)	Membrane Description	Membrane Thickness, mm	Temperature, °C	Current Density at 0.5 volts, ma/cm ²	Voltage ^(c) at 30 ma/cm ² , volts	Fuel Cell Resistance, ohms	Open Circuit Voltage, volts	Time from Start of Run, hours	Time of Run, hours
16	AA-1 with 50/ 50 Pt - Pd black	Impregnated with 50/50 Pt, Pd black; 20% in outer one-third layers.	1.03	65 ± 3	63.5 69.5 67.5 60.8 58.5	0.682 0.696 0.691 0.659 0.625	0.27 0.25 0.25 9.25 0.22	0.925 0.930 0.920 0.827 -	24 40 65 164 238	303
17	AA-1 with 50/ 50 Pt - Pd black	Impregnated with 50/50 Pt, Pd black; 20% in outer one-twentieth layers.	0.87	66 ± 2	-	0.670	-	-	7	7(b)
18	AA-1 with 50/ 50 Pt - Ir black on H ₂ side and Pt black on O ₂ side	Impregnated with 50/50 Pt, Ir black on H ₂ side, Pt black on O ₂ side; 20% in outer one- twentieth layers.	0.98	66 ± 1	-	0.585	-	-	2	7(b)
19	AA-1 with 50/ 50 Ag - Pd black H ₂ side and Pt black on O ₂ side	Impregnated with 50/50 Ag, Pd black on O ₂ side, Pt black on H ₂ side; 20% in outer one-twentieth layers.	0.80	63 ± 2	13.7	-	-	-	2	3(b)

(a) Refers to American Cyanamid Type AA-1 electrode with indicated type of catalyst powder sprinkled on membrane surface during water assembly.

(b) Terminated after indicated number of hours because of diminishing performance.

(c) Add 0.03 volts for leads correction.

TABLE IV

COMPARISON OF RESISTANCE AT $65 \pm 2^\circ\text{C}$ AND VARIOUS TIME INTERVALS FOR 20 PERCENT PLATINUM AND PALLADIUM BLACK CATALYST AND A 50/50 RATIO OF PLATINUM TO PALLADIUM-BLACK CATALYST IMPREGNATED IN THE OUTER ONE-THIRD LAYERS

		<u>Resistance (OHMS) at $65 \pm 2^\circ\text{C}$</u>					
<u>Time (Hours)</u>	<u>Catalyst</u>	<u>0-30</u>	<u>30-40</u>	<u>40-50</u>	<u>50-60</u>	<u>60-70</u>	<u>70-80</u>
<u>% Pt.</u>	<u>Test No.</u>						
20	(a)	.34	---	.37	.40	---	---
<u>% Pd.</u>							
20	(b)	.41	---	.33	.33	---	.35
<u>%(50/50)Pt. /Pd.</u>							
20	16	.27	---	.25	---	.25	---

(a) From Reference 1 - Fuel Cell Test No. 19 and 23

(b) From Reference 1 - Fuel Cell Test No. 20

TABLE V

SUMMARY OF FUEL CELL TEST RESULTS OBTAINED WITH VARIOUS
TANTALUM SCREEN ELECTRODES WITH C200B MEMBRANE

Fuel Cell Test No.	Electrode Description	Membrane Thickness, mm	Temperature, °C	Current Density at 0.5 volts, ma/cm ²	Voltage ^(b) at 30 ma/cm ² , volts	Fuel Cell Resistance, ohms	Open Circuit Voltage, volts	Time from Start of Run, hours	Time (a) of Run, hours
20	50 mesh Ta screen, impregnated with Teflon and Pt black by sintering, 22 mg Pt/cm ² , 5% Teflon	0.43	65±1	48.0	0.615	0.31	0.940	4	23
21	Same as Test 20 30 mg Pt/cm ² , 5% Teflon	0.56	65±3	-	0.635	-	0.910	7	7
22	50 mesh Ta screen, impregnated with a Pt black/Teflon "paste," with a thin Teflon film on the gas side. 20 mg Pt/cm ² , 6% Teflon	0.52	64±1	-	0.605	-	-	15	17
23	Same as Test 22. 30 mg Pt/cm ² , 5% Teflon	0.51	59±3	-	0.695	-	0.985	16	19
24	50 mesh Ta screen, impregnated with a Pt black/silicone "paste," 15 mg Pt/cm ² , 4% silicone	0.81	67±1	- 50.0	0.531 0.620	- -	0.875 0.930	41 45	66
25	50 mesh Ta screen, electroplated with Pt and waterproofed with silane 13 mg Pt/cm ² , 0.5% silane	0.90	65±1	- - -	0.508 0.520 0.550	- - -	0.909 0.923 -	7 22 23	51

(a) Tests were terminated at indicated number of hours because of diminished performance.

(b) Add 0.03 volts for leads correction.

TABLE VI

SUMMARY OF FUEL CELL TEST RESULTS OBTAINED WITH VARIOUS
TANTALUM AND STAINLESS STEEL SCREEN ELECTRODES WITH
C200B-PLATINUM IMPREGNATED MEMBRANES

Fuel Cell Test No.	Electrode Description	Membrane, Description	Membrane Thickness, mm	Temperature, °C	Current Density at 0.5 volts, ma/cm ²	Voltage ^(b) at 30 ma/cm ² , volts	Fuel Cell Resistance, ohms	Open Circuit Voltage, volts	Time from Start of Run, hours	Time (a) of Run, hours
26	50 mesh Ta screen, impregnated with Teflon & Pt black by sintering at 350°C 15 mg Pt/cm ² , 5% Teflon	Impregnated with Pt black; 20% in outer one-twentieth layers.	0.67	66±2	43.0 54.2 - 40.0	0.585 0.638 0.682 0.575	0.32 0.28 - 0.37	0.892 0.883 - 0.840	76 94 98 167	168
27	50 mesh Ta screen, impregnated with a Pt black/Teflon "paste," with a thin Teflon film on the gas side. 40 mg Pt/cm ²	Same as Test 26	0.95	66±1	2.2	-	-	0.710	1	6
28	Same as Test 27, but without Teflon film. 40 mg Pt/cm ² , 10% Teflon	Same as Test 26	0.90	65±2	18.0	0.310	-	0.910	23	24
29	50 mesh Ta screen, electroplated with Pt and waterproofed with silicone. 15 mg Pt/cm ² 1% silicone	Impregnated with Pt black; 20% in outer one-third layers	0.62	65±1	- - - - 45.0 45.0 45.0	0.550 0.555 0.560 0.590 0.590 0.607 0.605	- - - - - - -	0.793 0.782 0.850 0.820 0.800 0.810 0.817	16 40 65 88 138 163 187	192
30	Same as Test 29, but waterproofed with Teflon. 9 mg Pt/cm ² 7% Teflon	Impregnated with Pt black; 20% in outer one-twentieth layers.	0.91	87±1 100±1	- -	0.522 0.567	- -	0.922 0.835	119 136	138
31	160 mesh Stainless Steel (Type 304) screen, electroplated with Pt and waterproofed with silicone. 5 mg Pt/cm ² 2% silicone	Same as Test 30	0.83	65±2	32.0 41.5 44.0	0.518 0.592 0.600	0.47 0.40 0.35	0.975 0.980 1.000	42 66 89	185

(a) Tests were terminated at indicated number of hours because of diminished performance.

(b) Add 0.03 volts for leads correction.

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